Conceptual Design and Characterization of Composite Systems for Housing

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Aos meus pais,
mana,
e avó.

"It is only in a shared belief and insistence that there are practical alternatives that the balance of forces and chances begins to alter. Once the inevitabilities are challenged, we begin gathering our resources for a journey of hope. If there are no easy answers there are still available discoverable hard answers, and it is these that we can now learn to make and share. This has been, from the beginning, the sense and the impulse of the long revolution."

Raymond Williams

Resumo

Este projeto visa a criação e caracterização de um sistema compósito concebido a partir de uma base onde se tem em consideração fatores como o reaproveitamento de materiais de desperdício e os recursos serem provenientes de fontes renováveis.

Para a elaboração desde projeto foram reutilizadas fontes de desperdício florestal e desperdícios de plástico. Encontrando, deste modo, uma solução mais sustentável e ecológica, visando a área da construção ou outras aplicações de produtos de valor acrescentado, está-se simultaneamente a promover a limpeza das florestas e recolha de biomassa florestal e a contribuir para a redução do risco de incêndios florestais.

Respondendo também ao Projeto Floresta e Biomassa, no qual o objetivo é agir localmente de forma a encontrar soluções para problemas ecológicos específicos, nomeadamente incentivos à limpeza florestal, este projeto promove uma forma de valorização de desperdícios florestais para produtos ecológicos de valor acrescentado para reduzir o risco de incêndio

Neste sentido, foi concebido um material compósito constituído por uma matriz de policloreto de vinil (PVC) reciclado e fibras de eucalipto., da espécie *Eucalyptus globulus*. Para caracterizar este compósito e a sua própria matriz, que o ainda não estava, foram realizados provetes de prova posteriormente ensaiados nos laboratórios da FEUP através de: ensaios mecânicos, térmicos e acústica.

A seleção dos materiais sintéticos é fundamentada com o facto de serem reciclados, o que constitui um ponto-chave sob o ponto de vista ecológico, e o facto de provirem de empresas portuguesas, e a seleção dos materiais naturais como reforço prende-se com o facto de serem renováveis, biodegradáveis, abundantes em Portugal, e mecanicamente promissores. Os fornecedores de ambos os materiais são empresas portuguesas.

Abstract

This project aims to create and characterize a composite designed from a base that takes into account factors such as the recycling and renewability of the material resources.

In this project, it was seen an opportunity on the reutilization of the forest waste and waste of plastics. By creating a more sustainable and ecological solution, regarding the field of construction or further applications in other areas with value added products, this solution is promoting the cleaning of the wasted biomass on fields and, therefore, the forest cleaning which will contribute to reduce the fire risk.

To respond to the Forest and Biomass Project, which objective is to act locally to improve solutions for specific forest ecological problems, this project provides what could be the means to forest products and by-products valorisation as a tool to reduce fire risk.

Following this path, a composite made with recycled polyvinylchloride matrix filled with *Eucalyptus globulus* fibres was prepared. To characterize the behaviour of the so designed composite system and its subsequent applications, samples were prepared and specimens machined for testing. Experiments have been carried out at FEUP laboratories, such as flexion, tensile, compression, impact, reaction to fire, acoustic, water absorption and dilatometry tests.

The selection of synthetic materials is justified on the fact that they are recycled, which is a key point from an ecological point of view, and the selection of the natural fibres concerns the fact that they are a renewable, biodegradable, cheap, easily accessible, abundant source in Portugal and mechanically promising. Portuguese companies supplied both materials.

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À Maria Gabriel, e a todos aqueles que sempre me incentivaram a terminar este projeto, meus caros e amigos e família, obrigada.

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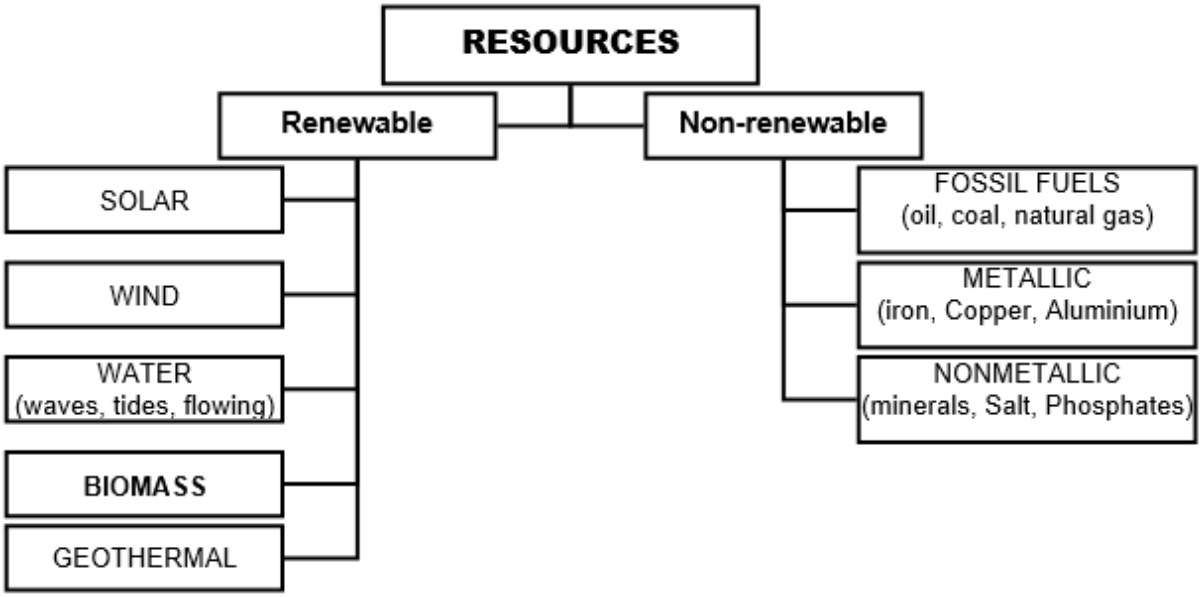
1. INTRODUCTION

1.1. Background and motivation

Resources

The 8th of August of 2016 was the last "Earth overshoot day", meaning that the resources that the planet can renew in a year have reached its limits, so the planet is literally living "credit" until December 31st of 2016, according to NGO Global Footprint Network. The expectations are that every year this day comes earlier. The very high consumption of products derived from non-renewable resources has been a great concern all over the world, once the humanity has already reached the limits of the Earth.

Also, the non-environmentally friendly life cycles of products and the "throwaway society" of today, constitute a dramatic source of waste grow, leading to accumulation of non-biodegradable refuse products, mostly plastic, in landfills when they reach their end of life time. Landfills have terrible and numerous impacts, such as costly conveyance of trash and access roads rapidly damaged by the heavy vehicles, scavengers[1] being killed, nuisances such as pests, dust, noise and odours. Besides those inconveniences, there is one rather large impact that landfills have got: these cause pollution to the local environment by contaminating the soil, the groundwater and aquifers, and producing methane[2] (Live Life Green 2009). The direct effects of landfills on soils and water, atmosphere, fauna and flora and consequently the human life leads to an urgent necessity to avoid this decadent tendency, and most important: solve the problem from its beginning by creating eco-friendly designing, from renewable resources.



Graphic 1 Examples of renewable and non-renewable natural resources

[1] An animal or other organism that feeds on decaying organic matter or refuse; a person who searches through and collects items from discarded material; a person employed to clean the streets.

[2] CH_4 – a greenhouse gases (GHG) 21 times more powerful and trapping 84 times more heat than CO_2. According to the Intergovernmental Panel on Climate Change (IPCC) scientists were more than 95% certain that global warming is mostly being caused by increasing concentrations of GHG.

Individuals, cooperatives and industries are all responsible for the researching of alternative solutions: less pollutant, ecological, more recyclable materials and designs using renewable sources of raw materials.

On the following topics are the materials that are part of the spur of this project.

Biomass

Sources of bioenergy called "biomass" include agricultural and forestry residues, municipal solid wastes, industrial wastes, and terrestrial and aquatic crops grown solely for energy purposes. Hence, bioenergy is the energy generated from the organic material of living or recently deceased plants or animals. If are considered as plant-based materials, it is called lignocellulosic biomass. Biomass is a good petroleum alternative once it is a renewable resource that is more squarely distributed over the planet than finite energy sources, and may be exploited using more environmentally friendly technologies. Also very important is the carbon neutral exchange of biomass, meaning the carbon absorbed during the lifetime of the organisms from which is created counters the carbon released by the combustion of the biofuel. Today, biomass resources are used via combustion to produce heat, to generate electricity and power, and to produce biodiesel and ethanol, or to process new bio-based polymers. Actually, among the natural fibre reinforced polymer composites, cellulose-based polymer composites have emerged as a potential environmentally friendly and cost-effective alternative to synthetic fibre reinforced composites.

Forest waste

Cellulose industry harvests the trees, mainly eucalyptus trees (the most abundant specie in Portugal), and often leaves at the field the small poles, branches, barks and leafs, which are considered no-value products, except for biomass energy production. Those, concurrently with the broom species, also very abundant in Portugal and also considered not to have economic value, create conditions to be a strong natural fire propagator. To appraise feasible solutions such as creation of value-added products, i.e. composite materials reinforced or filled with natural fibres for bio-polymers to substitute plastics derived from fossil fuels.

Plastic

Due to its hazards factors, such as the presence of chlorine, PVC is sometimes not even put under consideration when it is about to recycle. However, it needs to be recycled like all the other materials. Findings to new applications for this material will ultimately increase its recycling and selling.

Rubber

One specific problem of our so called "throwaway society" (Gartner 2015) of not recycling non-biodegradable waste is the stockpiles of tyres left in fields and forests, all over the world. The widely different chemical and ecologically problematic components and the cross-linked structures make tyres highly resistant to biodegradation. They are also easily inflammable and can trap methane gases, causing air, surface, ground water and soil contamination. The large volume produced annually - 1.4 billion units - and their cheap availability, places them among the most problematic sources of waste, that led to a 4 billion uselessly stockpiled tyre dumps (Sienkiewicz et al. 2012). It is established an urgent necessity to turn around this outrage to Earth. In fact, among their characteristics are their bulk and resilience, which make them attractive targets for recycling (Myhre and MacKillop 2002).

1.2. Objectives

In order to satisfy the increasing demand for new eco-designed products with higher performances, eco-friendly alternative materials need to be explored. Starting with renewable sources, the focus on replacing synthetic fibres boosters by natural fibres is considered with primary interest.

The spur of the present project follows this path: focusing specifically on natural vegetable fibres derived from forest waste, and recycled materials, the objective of this project is the conceptual design and mechanical characterization of a new bio-eco-composite system to improve what could be the means to forest products and by-products valorisation as a tool to reduce fire risk. While responding to specific environmental problems, such as the fires, it creates a possible new source of income.

1.3. Structure of the dissertation

The first chapter is the introduction where the background, motivation, objectives and structure are presented.

The second chapter is the state of the art where the background is deeply descripted to better understanding the pertinence of the subject of this project. The themes discussed are the composite systems and sustainable development, the use of natural materials on composites and the particular case of the eucalyptus, processing methods of composite materials and their recycling.

The third chapter is the description of the materials used to make the composite and the respective companies with which the development process was carried out.

The fourth chapter is the procedure explanation where the assembly of the sample's material to machine the test specimens is described

The fifth chapter is about the tests realized to obtain some properties and characteristics of the so designed composite system.

The sixth chapter is about the conclusions and future approaches.

The seven chapter presents the references.

2. STATE OF THE ART

2.1. Sustainable development

2.1.1. Development

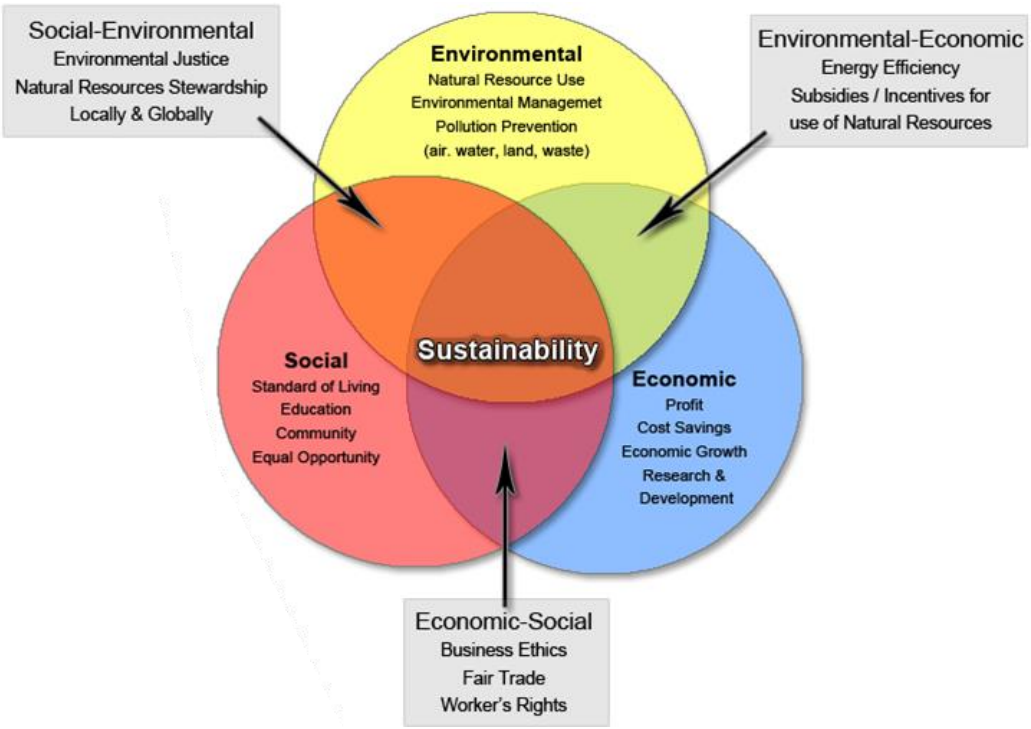
Development is a multi-dimensional process involving reorganization, restructuration and reorientation of the so-current economic and social system. Development is the process of improving the quality of everything, from products or services to the lives of human and all living beings.

Development is sometimes directly related to modernization – it emphasizes the process of change on social and political processes, which are required to produce economic advancement, hence leading to development – a better state of being.

Development in its way to improve people's life is not a fully truly achievement, neither a great advancement, if it is not durable and likely to achieve lasting satisfaction of human needs. So, development should sustain as long the new improved state of things interact in harmony with earth and every kind of life. It means that development is a process that ought to be temporally efficient and transversal to the globe to be considered as well. The implemented development appearing as new status with new tools should be enough to adapt to new circumstances. It is a concept that starts early in the process of product conception.

2.1.2. Sustainability

Sustainability has got three main pillars that are interdependent and mutually reinforcing and not mutually exclusive. They are economical sustainability, environmental sustainability, social sustainability and, as seen on Graphic 2.



Graphic 2 Venn diagram representing the three spheres of sustainability (Sustainability Matters Blog, 2013)

Environmental sustainability reflects the full understanding of the natural gentle flow of the Earth's natural resources to resettle, and its eventual scarcity. Among other unsustainable attitudes, we, as human beings - the unique responsible ones for every problem in the world - are globally extracting natural resources faster than the earth can replenish.

The concept of resource efficiency may consign sustainability. Resource efficiency is about the reduction of the environmental impact from the raw material extraction, to later use and latest disposal, passing through the production and consumption. Resource efficiency comprehends that the current, global, economic growth and development cannot be sustained with the current production and consumption patterns (Mark Hyman 2013).

Social sustainability is the gentle interaction between the environmental and economic sustainability and economical sustainability should beneficiate from the environmental sustainability through a circular economy.

2.1.3. Sustainable Development

The sustainable development is a concept transversal to environment, economy and social issues (Ferrão 2012). This concept found its success and international relevance after the World Commission on Environment and Development (Brundtland Commission) published its report in 1987, presenting the most famous definition of the so new concept: "Sustainable development is a development which meets the needs of current generations without compromising the ability of future generations to meet their own needs" (Kaj Bärlund 2016).

2.1.4. Education and sustainable development

The United Nations Decade for Education for Sustainable Development (ESD), initiated in 2005, demonstrates the great influence of education for sustainable development. Education, in its all transversal areas, from humanities to art or engineering, has a role of importance on transmitting the sustainable development matter to all ordinary citizens as (or not) future decision-makers.

ESD promotes the sharing of knowledge to every human being, from learner to teacher, for having the necessary competences to act sustainably today, led by the will of shaping a sustainable future. By including into both teaching and learning the basic fundamental subjects about the sustainable development issue, such as the climate change and disaster risk reduction, sustainable consumption strategies, biodiversity or poverty reduction, it stimulates competencies like critical thinking about the today's decisions of upper commissions and governments on what concerns a sustainable future, and the making of decisions in a collaborative way. This noble focus requires the inclusion of participatory and interacting teaching and learning methods that allow the individuals to acquire values and motivations, empowering them to change their daily behaviour and take action for a sustainable future (© UNESCO 2014).

For this simple but powerful purpose, education requires far-reaching changes in the way it is often practised today. Sustainable development absolutely needs democratic thinking, but then again it can also help strengthen democratic institutions through consensus-based public participation.

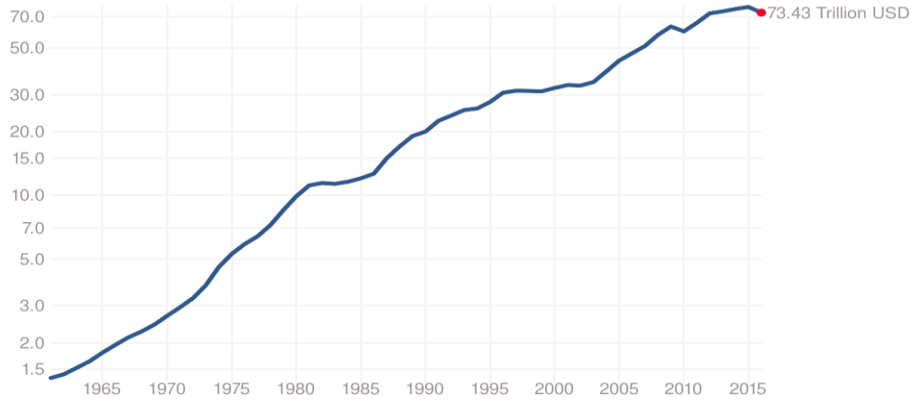
2.1.5. Models of economy

Linear Economy

The last century of industrial evolution has been following a one-way model of production and consumption: the linear model of production, or linear economy. Raw materials are collected, goods are manufactured, later they are sold and used, and after being used, those goods are discarded or incinerated as waste, as it can be observed on Figure 1.

Figure 1 Linear economy model (adapted from The Value Loop Economy, 2010)

Apparently, the system had been successful in terms of providing affordable products to consumers, as can be seen on Graphic 3, the global GDP[3] has been growing since ever, and created hitherto unknown levels of material welfare. Even though the economic downturn provoked a great depression reducing the demand, since 2009 resource prices have recovered faster than global economic output.



Graphic 3 Global GDP along time (WBG 2014)

Within this linear model, resources needed for production and consumption are extracted from Earth as faster as possible, in order to respond to the demand on the best time, whenever and wherever it is geographically and economically possible. Yet, this approach is creating a world of soon 9 billion consumers (Ranjani 2014) who are actively buying manufactured goods within a one-way track linear model with no plans for reuse or active regeneration of the natural systems from which they have been taken.

The linear model worked perfectly when resources were plentiful and waste was thoughtless to dispose. Today, with the continuous growth in population, GDP and consumption, climate change getting worst, regulation and cutting edge technology improved each day, there is a direct moral motivation but also economic incentives to rethink the old practices of "take-make-dispose" consumption habits. Those have been working since the 1760's (Export Leadership Forum 2015) and are still followed by most supply chains.

The current linear "take-make-dispose" economy came to a point where it approached results in massive waste with worrisome consequences, reaching its limits. According to the book "*Rubbish!"* (Girling 2005) 90% of the raw materials used in manufacturing become waste before the product leaves the factory, while 80% of products made get thrown away within the first six months of their life. This, coupled with growing tensions around geopolitics and supply

[3] Gross Domestic Product (GDP): a monetary measure of all final goods and services produced in a time period

risk, is contributing to volatile commodity prices. This will threaten and encumber companies, hinder the world

A circular economy is an attractive and viable alternative, presented as a functional economic model that can be a real solution to the planet's emerging resource problems and landfill disposals. Business has already started exploring it.

Circular Economy

The circular economy is a redesign of our current production and consumption model by seeing waste as resource and an opportunity. Circular economy suggestion is by inducting an eco-friendly solution to industrial economy by bringing back into the supply chain the products after their end of life, as a resource. It envisions no waste. It means that reuse rather than disposing closes the loop, as seen on Figure 2. Besides benefiting the economy by producing no waste, it also means no pollution. For that reason, circular economy is a model that promotes a sustainable economic flow whilst improves environmental sustainability.

Figure 2 Circular economy model (Pre-sustainability, “Complementing The Circular Economy with LCA”, website acceded 12/09/2016)

The concept distinguishes two types of material flows: technical and biological cycles. In the bio-cycle, all life processes regenerate discarded materials and resources despite or without human intervention. It is prepared to re-enter the biosphere safely. In the technical cycle, materials are recovered and restored by human intervention, that recreates order in any timescale considered.

Following there are some principles and characteristics (Ellen MacArthur Foundation 2015):

- Eradicates waste and optimise resource yield through careful design: products are designed thoughtfully to fit a cycle for remanufactured, disassembly or repurposing to keep components and materials circulating in and contributing to the economy and avoiding the generation of waste;
- Aims to run on renewable energy, renewable sources of energy and raw materials;
- Minimises, tracks, and hopefully eliminates the use of toxic chemicals;

- Thinks in systems: the ability to understand how parts influence one another within a whole, and the relationship of the whole to the parts, is crucial;
- Thinks in cascades: the opportunity to extract additional value from products and materials is the essence of value creation, by cascading bio materials through more applications;
- Thinks in modulus: modularity, versatility and adaptability together build the resilience necessary to face unexpected external changes or internal problems;
- Preserves and enhances natural capital and fosters system effectiveness: dematerialising utility delivering it virtually whenever possible, selecting the resources technologies and processes wisely, and reducing damage to human utility, such as food, mobility, shelter, education, health, and entertainment, and managing externalities, such as land use, air, water and noise pollution, release of toxic substances and climate change.

Then, once recycling offers limited appeal as its processes are energy-intensive and can downgrade materials, ultimately leading to demand for virgin materials, the circular economy goes beyond recycling and reusing. Based around a restorative industrial system, it is geared towards designing out from waste. The graphic shown on Figure 3, shows how recycling is an 'outer circle' of the circular economy, requiring more energy input than the 'inner circles' of repair, reuse and remanufacture.

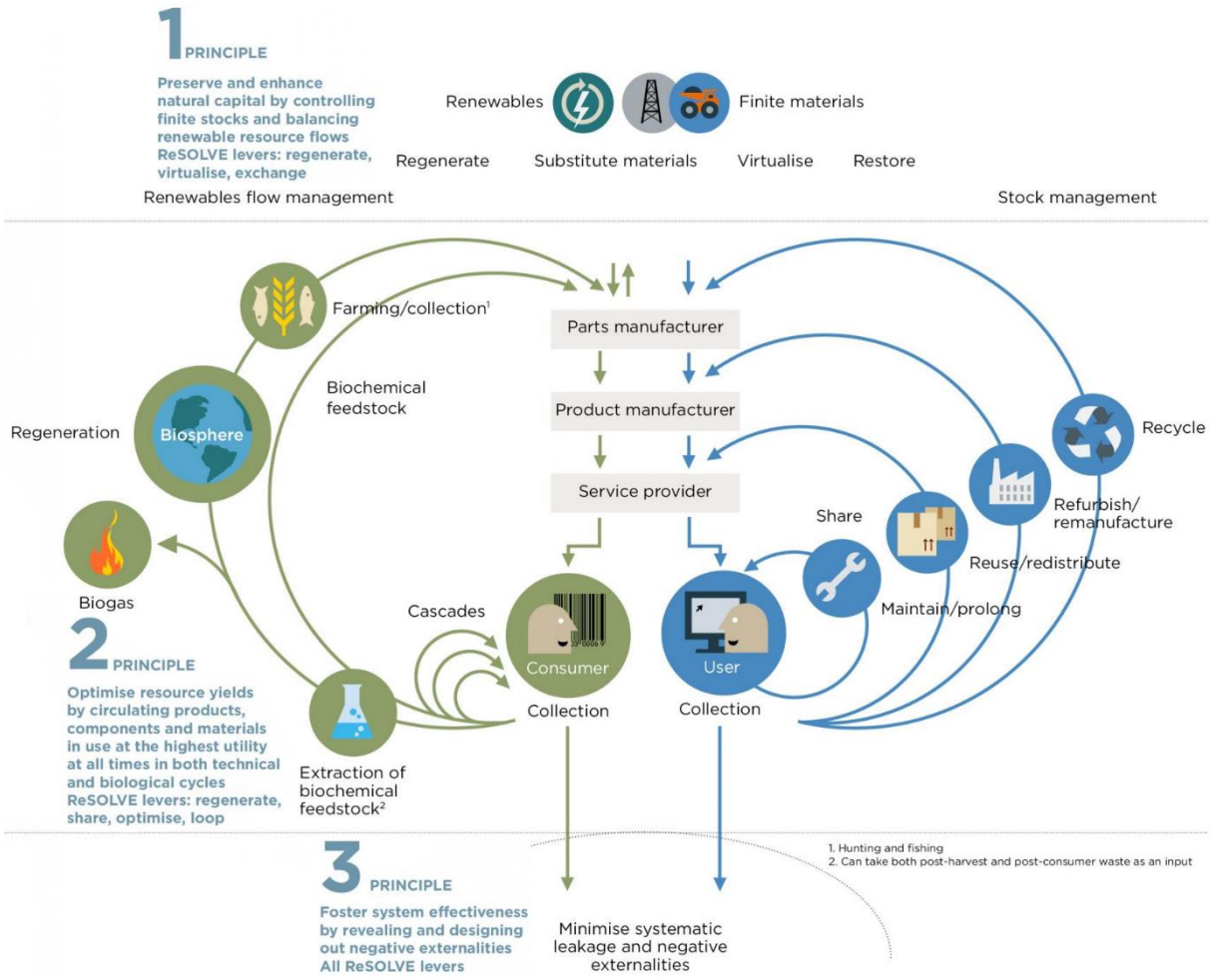


Figure 3 Outline for a circular economy (Ellen MacArthur Foundation 2015)

Besides the eco-design, it is important, in accordance with the waste hierarchy, and for the purpose of reduction of greenhouse gas emissions originated from waste disposal on landfills, to simplify the separate collection and proper treatment of bio-waste in order to produce environmentally safe compost and other bio-waste based materials.

As seen, the circular economy aims to eradicate waste right from the manufacturing processes methods, as lean management aspires to do, but never forgets the impact of the material itself, throughout the life cycles of products, and their components and their use and reuse. This aided by smart product design, help define the concept of a circular economy. It is restorative and regenerative by principle, seeking to rebuild capital, whether this is financial, manufactured, human, social or natural. This ensures enhanced flows of goods and services.

The Europe 2020 strategy for smart and sustainable growth established that reaching circular economy was the major part of the resource efficiency agenda, expecting on that date recycling should be on 50%, the circular economy releases 2 million jobs on Europe and that productivity from resources increase 30% until 2030. Circular economy could increase companies' profits by over 50% and reduce waste by over 95%. Some experts say that if circular economy is not already on one company, it will be within the next 3 – 5 years, predicting it will be the greatest shift in production and consumption since the start of the Industrial Revolution. (Export Leadership Forum 2015).

For the companies to know how much advanced they are on their transition from linear to circular economy, The Circularity Indicators Project provides companies with methodology and tools that allows them to assess and estimate how well a product performs in the context of a circular economy. These indicators consist of both a Material Circularity Indicator that measures how restorative the material flows of a product or company are, and complementary indicators that allow additional impacts and risks to be taken into account. The full Circularity Indicators methodology, together with a project overview report and a collection of non-technical case studies, are available for download.

Members of the European Commission and the aerospace, automotive, shipbuilding, rail, construction sectors intend to outline a strategy for transitioning from a linear to a circular economy. While the challenge seems difficult, it can be motivating to hear the success stories of others as well as learn from the challenges they faced along the way. There are many companies that have already made the transition from linear to circular economy, all of them with benefits on profits and savings:

- Circular economy design: Bralform, Evocative, Agency of Design, Active Disassembly;
- New business models: Bundles, Philips & Turntoo, Splosh, Desso;
- Reverse cycles: Re-Tek, Mazuma Mobile, Caterpillar, Rype Office

The circular economy is a framework that draws upon and encompasses principles from biomimicry, industrial ecology or blue economy. There are also other relating's to this model i.e. cradle to cradle or upcyling, explained on the following (Shilling 2013):

- **Biomimicry** – A name coined by Otto Schmitt in the 1950s, is a discipline that observes and studies the nature's processes, and then transfer the ideas and analogues from biology to technology to solve human problems, (Vincent et al. 2006);
- **Industrial Ecology** – Sometimes considered as the science of sustainability, is the study of material and energy flows through industrial systems;

- **Blue Economy** – Initiated by Former Ecover CEO and Belgian businessman Gunter Pauli, states *"using the resources available in cascading systems, the waste of one product becomes the input to create a new cash flow";*
- **Cradle to cradle system** – Created by Michael Braungart and American architect Bill Mc Donough, it considers that all material involved in industrial and commercial processes can be seen as nutrients, of which there are two main categories: technical and biological. Technical nutrients should include only materials that do not have a negative impact on the environment. Biological nutrients are organic and can be returned to the soil without specific treatment to decompose and eventually become food for the ecosystem; so, it means designing products so when they are at the end of their useful lives their components can be used for some other productive purpose that doesn't negatively affect the environment;
- **Upcycling** – refers to the reuse of objects or materials that are at the stage of being discarded, but which can be turned into new value products;
- **Cradle to grave** – this means the product is eventually left in the environment with no useful means and/or negative consequences, i.e., as hazardous waste.

According to the latest report by the World Economic Forum and Ellen MacArthur Foundation, with analytical support from McKinsey & Company, applying circular economy principles to global plastic packaging flows could considerably reduce negative externalities such as leakage into oceans. Given projected growth in consumption in a "business-as-usual scenario", by 2050 oceans are expected to contain more plastics than fish (by weight), and the entire plastics industry will consume 20% of total oil production, and 15% of the annual carbon budget (Ellen MacArthur Foundation 2015). The report New Plastics Economy: *Rethinking the future of plastics*, that aims to accelerate business-driven innovations to help scale the circular economy, provides a vision of a global economy in which plastics never become waste, and outlines concrete steps towards achieving the systemic shift needed.

Case Study of circular economy: A'gua bottle,

Donald Thomson, a Canadian constructor and self-taught entrepreneur have started giving lessons on a music school to children. Once, he had organized a beach cleaning with his students on Costa Rica and observed that when plastic bottles were flattened and put in line they looked like slate tiles. From hete began a long journey that is called today "Bottle-to-tile project". Thomson created a new company - Center for Regenerative Design and Collaboration – that began to work "backwards". They first made the tile design, and then thought about what was the shape of the bottle needed to do it quickly, which ultimately led to the design of the so-called A'Gua bottle. This bottle is made from 100% plastic recycled and the water comes from Juan Blanco National Park. In conclusion this project recycles plastic bottles into a new product that is a tile of a roof. This transformation is possible due to an eco-design of the original product, needed for adaptation and creation of another product with a long life cycle (up to 50 years), as seen on Figure 4. The colouring that fulfills the bottle can be made of wasted plastic, card, papers and porous cement. At the end of its life, the plastic tile can be recycled again. The bottles are easily implemented to a system that is locally applied in roofs or the chosen coverage by unrolling a scroll of tiles previously fixed on a roll, and can be assembled 400 bottles per minute, as seen on Figure 5. (Pais, Diana and Luís Pinto, 2015)**.**

(a) (b)

Figure 4 Initial water bottle and the final tile obtained (a) and flatted full coloured water bottle (b)

Figure 5 System of locally application of the cover

2.1.6. Waste Management

Waste Management is the process required to manage waste from its inception to its final disposal. All processes and resources for proper handling of waste materials, from the collection, transportation to dumping facilities in compliance with health codes and environmental regulations, disposal of waste products.

It can be considered a policy which aim is mostly radicalize the problem of waste generation from its core by worrying about avoiding the waste stream, instead of giving solutions to it after it's done. It means stopping the waste stream from the very beginning, reducing the use of resources and favour the practical application of the waste hierarchy.

The central principles of waste management lead to specialised concepts:

- Waste hierarchy;
- Life cycle assessment (LCA);
- Resource efficiency;
- 3 R's: reduce, re-use, recycle.

The EU Waste Management Hierarchy[4] is usually presented diagrammatically and indicates by a descendent order of preference the most preferred action to reduce and manage waste, as shown on Figure 6, the waste hierarchy is presented as an inverted pyramid.

Figure 6 The Waste Management Hierarchy Diagram (Acceleratio, Waste & Resource Management, accessed 12/09/2016)

Therefore, at the top of the pyramid representing the essential thrust of the policy is to take action first. Occupying the bigger and most important step is the avoidance of the waste generation. This step refers to the prevention that must be towards the waste generation, by avoiding it, e.g., buy loose food avoids and have a unique eco-plastic shopping bag[5]. It consists on the most efficient way to counteract every tendency of an emergent waste stream and will, ultimately, lead to conservation of resources.

The next preferred action is to reuse the waste. Go through the first step and the impossibility of avoiding completely the waste generation, individuals and industries should worry and gather their efforts towards the reduction of the waste generated through, e.g., re-use fitness. It consists on giving a new purpose to a product or its by-products when achieving the end of their life cycle. Actually, due to their long life cycles, composites often outlive as an upcycled product, rather than the one it was created for. That means composites are often reusable, depending on the product's flexibility, consumer's necessities, imagination and know-how.

Recycling, the third most favoured option to take, is any recovery operation by which waste materials are reprocessed into products, materials or substances whether for the original or other purposes. It includes composting or anaerobic digestion and it does not include incineration. The waste hierarchy strongly lies upon the "3 R's" reduce, reuse and recycle, which help hierarchize waste management strategies in a descendent order of their convenience (Romero 2016).

Following the downstream steps of waste hierarchy diagram, there is materials recovery as waste-to-energy. If energy is recovered from processes such as combustion or pyrolysis, or incineration from landfill, it also belongs at this level of the hierarchy. (Mark Hyman 2013).

Disposal of waste is the last and the least desirable choice, either in landfills or through incineration without energy recovery, especially due to their environmental hazardous effects and very low sustainable economic flow generation. This final step is a last resort for waste which has not been able to be prevented, diverted, or recovered in the preceding steps (Mark Hyman 2013).

[4] Different versions of the hierarchy have been adopted by different countries, although they are all broadly similar to that one referred on this document

[5] Several examples of eco-friendly plastic shopping bags

Ben Drogt (Biinc[6]) have truly shown how the reusing of materials can be applied to everything: in the Netherlands reusable wind turbine blades were upcycled to make playgrounds for children adding to the multiple uses for composite materials, as it can be seen on Figure 7 (EuCIA 2016).

Figure 7 Wind turbine blades upcycled to a playground at Netherlands (Denis Guzzo, NoTechMagazine)

Also at our daily lives there can be seen on internet do it yourself (DIY) great ideas with endless home made products reusing waste materials

2.1.7. Waste For Life

There are now some networks developed to respond to specific ecological problems, such as the *Waste For Life* (WFL) network. WFL is a network not interested in profit, led by a diverse group of people like scientists, engineers, educators, architects, artists, designers, and cooperatives, all working together to develop poverty-reducing solutions. By teaching communities how to deal with their local environmental problems regarding the garbage and the biggest sources of waste, WFL aims to is to provide access to technology and scientific knowledge for everyone, especially to non-privilege people. Based upon principals of solidarity, cooperative interchange, and social justice, WFL put impoverished communities in control to promote self-sufficiency, autonomy and economic security.

WFL uses scientific knowledge and low-threshold/high-impact technologies to add value to resources that are commonly considered harmful or without worth, but are often the source of livelihood for society's poorest members. Their twin goals are to reduce the damaging environmental impact of non-recycled plastic waste products and to promote self-sufficiency and economic security for at-risk populations who depend upon waste to survive. This double goal in which while they reduce the available waste, they transform it to create a source of income to the scavengers, is adapted locally to the kind of waste available, technologies needed and the will of population.

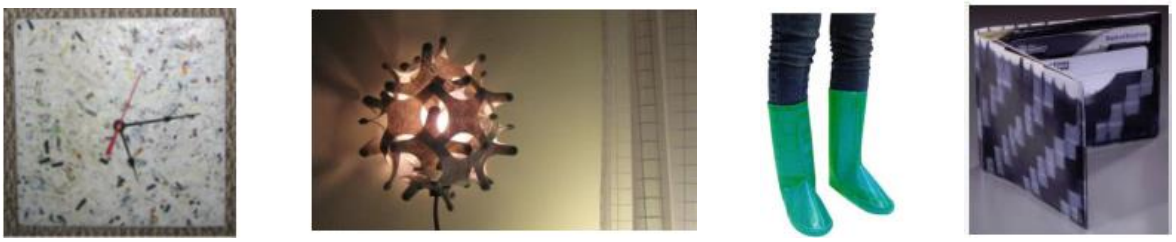
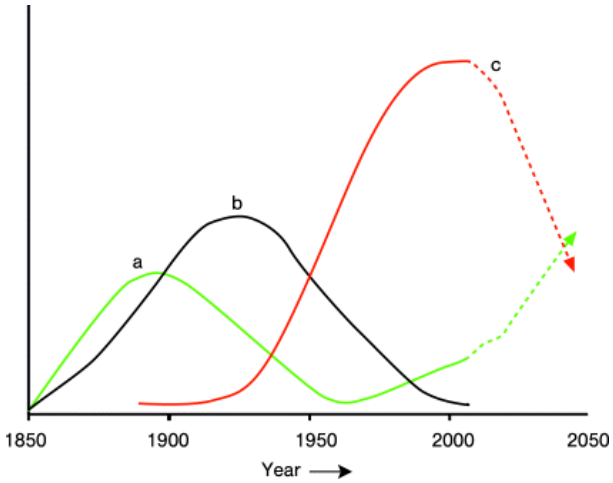


Figure 8 Products made by WFL workers, from discarded plastic and cardboard (wasteforlife.org, 2016)

[6] BiinC is a Dutch company (founder: Ben Drogt, 2010) that promotes composite materials as the material of choice regarding performance and sustainability in many different applications

2.2. Composites and sustainable development

The inevitable necessity of the changeover of the chemical industry to renewable feedstocks for obtaining raw materials is growing, because our fossil raw materials derived from prehistoric organic matter are irrevocably decreasing, and the pressure on our environment is intensifying. Even tough fossil oil will be around for a long time, the prevailing issue is when will be the end of cheap oil. Experts realistically predict this to occur within the next two to three decades. Accordingly, the curve for the utilization of biofeedstocks shown in Graphic 4 will have to rise that much that it meets that of fossil raw materials somewhere around 2030–2040.



Graphic 4 Raw materials used by the chemical industry in historical perspective: a) renewable feedstocks; b) coal; c) natural gas, oil. (Lichtenthaler 2010).

2.2.1. Composites

Composites are made from at least two materials that complement each other. The individual materials that constitute composites have significant different properties from each other. When these materials are combined, they create a composite material with characteristics that are better and different to the initial properties of the individual components. Better characteristics on the weight and mechanical strength are the most common ones.

A composite material can be defined as a combination of a matrix and a reinforcement. The reinforcement is used to fortify the matrix in terms of strength and stiffness. The matrix, normally a form of a resin, keeps the reinforcement in the desired orientation and protects it from environment and chemical attacks. The matrix also bonds the reinforcement so that applied loads can be effectively transferred to it.

On Figure 9, there is shown a representation of the five basic types of composites materials based on the kind of the reinforcement, and the respective representative aspects on materials.

The reinforcements are:

- Fibre;
- Particle;
- Laminar or layered;
- Flake;
- Filled composites.

Today, the most common reinforcement is the fibre. Fibres can be short, cut, long, aligned or placed in different ways to affect the properties of the resulting composite.

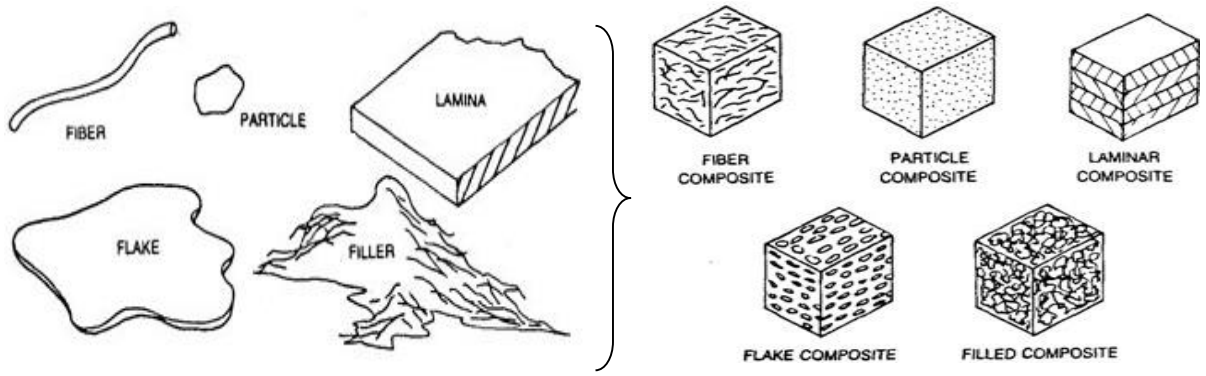


Figure 9 Classification of composites based on reinforcement, (RAJPUT et al. 2015)

Regarding the type of matrix, the classification of composites lies upon three main categories:

- Metal matrix composite;
- Ceramic matrix composite;
- Polymer matrix composite.

From all the thermoplastic polymers (TPS) used in the manufacture of composite materials employing natural fibres as reinforcement, standout polypropylene (PP), poly-ethylene (PE), polystyrene (PS), and polyvinyl chloride (PVC). From the thermosets polymeric matrices that are used in the manufacture of composite materials, standouts are epoxy, phenolic, and polyester resins. Both kinds on the petroleum-based polymers field. On chapter 2.2.5 2.2.5Natural Matrix, bio-composites natural matrices, biodegradable matrices, and from renewable resources are described.

Composite materials, acknowledged as 'space-age' materials, are finding their way to a great place in the world, being used on conveyance sector reducing the weight for increased fuel efficiency, replacing wood on places where weather and environment act scarcely, or in building systems. Composites have a long life, and for all stated they represent a very important role and enormous mass all around the world.

The European Composites Industry Association (EuCIA[7]) held a conference in Brussels on 19th of January of 2016 to examine the increasing role that composites will play in creating a more sustainable Europe. The members of the European Commission and the aerospace, automotive, shipbuilding, rail, construction sectors attended the event as the ECI intends to outline a strong strategy for transitioning from a linear to a circular economy (EcoComposites 2016).

The EcoComposites website is dedicated to provide technical and commercial news and features on new innovative composite materials that have a minimal impact on the environment by using renewable raw materials or are manufactured with as little environmental impact as possible. Ideally, these products should biodegrade at the end of their useful life or be able to be recycled and preferably, upcycled into useful new products.

[7] EuCIA is the Brussels based leading Association of the European Composites Industry, representing European National Composite Associations as well as Industry Specific Sector Groups, such as those targeting end segments like automotive or those promoting particular product groups or processes.

2.2.2. Bio-composites and Eco-composites

Composites may be synthetic or not. However, the development of high-performance materials made from natural resources is increasing worldwide. Due to environmental and sustainability issues, there are remarkable achievements in green technology in the field of materials' science, through the development of eco-composite, bio-composites and bio-industry.

Bio-composite and Ecocomposite

Bio-composite is a composite material where at least one of the constituents is derived from biomass, (Carruthers and Quarshie 2014), whether they are made of natural fibres with synthetic resins, natural resins with synthetic fibres, or both natural components.

An eco-composite may also be constituted by natural polymer, or be reinforced with natural fibres (Bogoeva-Gaceva et al. 2007).

Also, there are included on this nomination (Faruk et al. 2012):

- Composites from which the raw materials that are on their origin are renewable resources;
- Composites which waste can be managed within an eco-friendly way at the end of their life, like composting, biomethanation, recycling or other;
- Composites where the successive transformation processes from the raw materials to the final products are environmental-friendly: low energy consumption, low emissions.

Ecocomposite materials are presented with ecological advantages over conventional composites, such as the following benefits:

- Lower costs because of reduced cost of raw materials, smaller cycle times, lower weights, and reduction in the fuel consumption (vehicle parts);
- Identical mechanical properties of glass-reinforced parts, with fabrication advantages such as smaller tool wear, good sound insulation, and geometrical stability;
- Eco-friendliness, renewability of the raw materials, recyclability, no toxicity, and CO_2 neutrality

On Figure 10 is an example of how versatile bio-composites can be. They find applications in many fields from construction, automotive, biomedicine, goods, and much more.

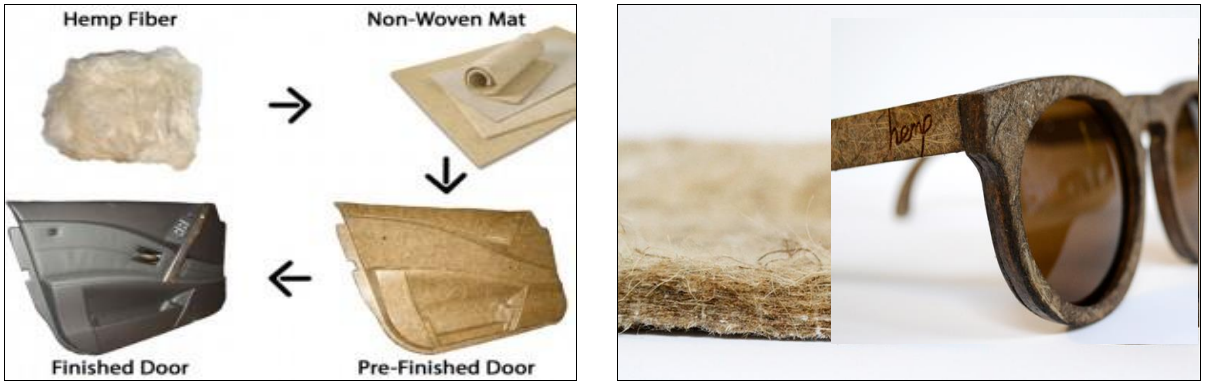


Figure 10 Examples of different bio-composites made with the same natural reinforcement: hemp

When one cares about ecology, this renewable resources and the ease on the composting or recycling of the materials win against the conventional construction materials and hence to construction and demolitions residues. In addition, they weigh less and are cheaper. Studies regarding their use as load-bearing components are also encouraging, (Taylor & Francis Groups 2016).

As an emerging material, measuring the impact of composites on the environment remains fundamental. There are multiple tools to help to transit to the circular economy, to have more sustainable actions, get to know our carbon footprint, get informed about the impact of our actions. It's up to each one to decide what option to take next. An example among many others is an innovative release of an application announced by Roberto Frassine, the (EuCIA 2016) president, EcoCalculator. It allows members to measure the environmental impact of composite materials without the need for Life Cycle Assessment (LCA) know-how. EcoCalculator creates a so-called EcoFactsheet for clients and stakeholders based on quality data and the reports generated by the tool will be compatible with the major analysis programs in the market

2.2.3. Wood and Wood polymer composite (WPC)

Hardwood and Softwood

The wood is often classified as either a hardwood or softwood, related with to its physical structure and so it is tempting to think of hardwoods as being hard and durable compared to soft and workable softwoods. However, is not necessarily like this, such as in the cases of wood from yew trees (a softwood that is relatively hard) and wood from balsa trees (a hardwood that is softer than softwoods). The distinction between hardwood and softwood actually has to do with plant reproduction. All trees reproduce by producing seeds, but the seed structure varies.

Hardwood trees are angiosperms: plants that produce seeds with some sort of covering, this might be a fruit, such as an apple, or a hard shell, such as an acorn.

Softwoods, on the other hand, are gymnosperms: plants that produce seeds with no covering. In conifers like pines, these seeds are released into the wind when they are mature. This spreads the plant's seed over a wider area.

Both hardwood and softwood unitary cells are tubular structures and can be pulped. The chemistry and fibre morphology of hardwood and softwood pulps are significantly different. The average length of hardwood and softwood fibres is approximately 1 and 3 mm, respectively. The width of the fibres may vary between 10 and 50 μm and the wall thickness between roughly 1 and 5 μm, (Santos et al. 2015).

However, the main differences that happen to be true between those two kinds of woods are presented on

Wood Polymer Composite

Wood fibre polymer composites, wood polymer composite or wood plastic composites (WPC) are composites that contain wood particles or other fibrous materials (even nonwood) combined with polymer resins (thermosets or thermoplastics). Some examples of utilised woods are shown on Figure 11.

Table 1 Main characteristics that differ hardwood from softwood differ

	SOFTWOOD	**HARDWOOD**
SHEDDING OF LEAVES	Angiosperm 	Gymnosperm 
	Coniferous trees: Needle-leaved evergreen tree: does not shed its leaves	Decidious trees: Broad-leaves tree: sheds its leaveas in autumn and winter
WOOD PROPERRTIES	**Softwood**	**Hardwood**
Colour	Lighter	Darker
Weight	Lighter	Heavier
Density	Low	High
Growth	Faster	Slower
Annual rings	Distinct	Indistinct
Fire resistance	Poor fire resistor	Good fire resistor
Endurance	Less durable	Highly durable
Ease on working	Easy to work with	Hard to work with
Heartwood & sap wood	Indistinguishable	Distinguishable
Resin content	Few soft wood are resins	Less
Price	Less expensive	More expensive
Environmental impact	Less environmental impact	More environmental impact

Figure 11 Some examples of wood to fulfil WPC: a) Wheat Straw; b) Bamboo; c) Rosewood; d) Olive Waste; e) Rice Husk; f) Hard Wood. (Rotexmaster)

The plastics PVC, PP and PE are the three most widely used thermoplastics on WPC. PVC provides the greatest strength and stiffness for WPC composites, followed by PE and PP.

Currently, WPCs have become good substitutes for solid wood, once they have similar appearance, are more durable while requiring little maintenance.

Plastic processors see wood as good and relatively inexpensive filler, readily available, that can lower resin costs, improve stiffness, increase profile extrusion rates, and act as an environmentally friendly method by which the use of petroleum-based plastics could be decreased. Forest product companies see waste wood and wood of low-commercial value as a source to produce WPCs and increase the added value for the utilisation of forest waste, (Taylor & Francis Groups 2016).

WPC permits more complex shapes in comparison to wood materials, because the principal manufacturing processes of WPCs are generally extrusion and injection moulding usually for PVC or PE matrix-based profiles, Injection moulding is finding its way on into furniture, technical parts, consumer goods and household electronics. Dashboards, car door panels and airline service trolleys are just more three of the various areas where bio-composites are now finding success in lightweight construction designs. More common applications for these composites are in decking, fences, railings and outdoor landscaping features, and other goods.as shown on Figure 12, as well as furniture and automotive products.

(a) (b)

Figure 12 Applications of WPC manufactured from granulated WPC: (a) containers made by injection moulding (FKuR, Fibrolon); (b) outdoor decking boards made by extrusion (ProTechWood)

Additives

PVC base polymers used in wood polymer composites for decking and railing systems can have additives to improve performance, for example: the mixture between PVC and a plasticizer is essential for good performance.

Other examples are PHA additives, that because of their miscibility with PVC, the PHA additives will not migrate out of the PVC and are easy to handle and process. The data generated by the company Metabolix, says that the addition of PHA i6003 during processing, even at a low level, improved wood filler incorporation and dispersion and made it possible to increase the proportion of wood flour used while decreasing the amount of PVC needed. Moreover, it acted as an efficient fusion promoter, reducing torque in the extrusion. For railing applications, the end-product also exhibited significantly improved mechanical properties and surface finish (Metabolix 2014).

Mundial Panorama (2012) (EcoComposites 2013)

According to a report by the Nova Institute, 350,000 tons of wood and natural fibre composites were produced in the European Union in 2012, representing between 10-15% of the total worldwide natural fibre composite market, that was around 2.5 million tons. Relatively to the wood-plastic composites (WPCs), the total volume produced in Europe was 260,000 tons from which around 90,000 tons of natural fibres composites for the automotive industry. China has the strongest growth rates, with a production volume of 900,000 tons in 2012 and Germany leads in terms of the number of manufacturers as well as in production volumes.

2.2.4. Natural fibres

Some decades ago, when thermoplastic replaced metal, their prices increased, which ultimately led to the need of including fillers to make compounds and reduce costs. Among those fillers there are the fibres that can be divided in two main groups: the man-made ones and natural ones.

The natural fibres can have animal origins, lignocellulosic basis or have mineral source. The lignocellulose natural fibres, on which relies the pertinence of this project, are often referred to as vegetable fibres. They are extracted from plants and classified into different categories, depending on the part of the plant they are extracted from, as it can be seen on Figure 13.

Natural cellulose/lignocellulose fibres can be used for composite materials as a reinforcement or filler, or they can be used as raw materials for cellulose production, often to produce the cellulose pulp being on the origins of cardboard and paper, as the fibres from wood. Bast fibres are generally found in the stems of plants. They provide the plant its strength and usually they run across the entire length of the stem, therefore they are long fibres. The fibres extracted from the leaves are rough and sturdy and form part of the plant's transportation system. On the other hand, fruit fibres, extracted from the fruits of the plant, are light and hairy, allowing the wind to carry the seeds. Agricultural fibres include crop residuals, such as straw, stems, hulls, and by products from wheat, corn, soybean, sorghum, oat, barley, rice, and other crops.

Advantages

There is a growing interest to use these fibres as fillers and/or reinforcements in plastics and composites. The main factors that make them attractive to manufacturers can be their flexibility during processing, highly specific stiffness, and low cost (on a volumetric basis) Additional advantages of the use of natural fibres in composites are:

- They have a low density;
- They are abundant and renewable;
- They are environmentally friendly.
- They have a high degree of flexibility during processing and generally do not damage the equipment;
- Their renewability, biodegradability, nontoxicity, and good insulation properties.

Also, compared with glass fibre composites, natural fibre composite have got advantages such as no abrasiveness to equipment and freedom from health problems due to skin irritation during handling and processing.

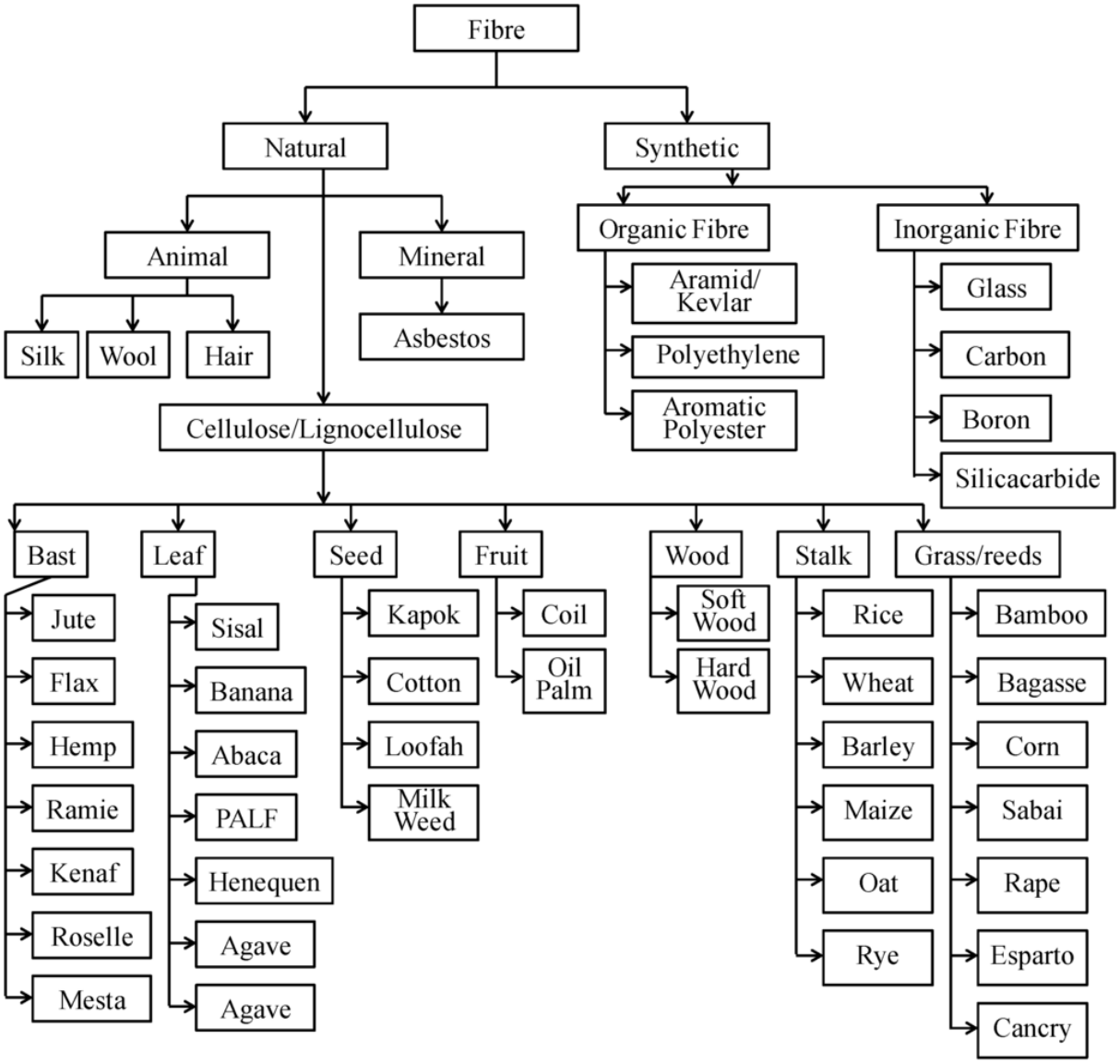


Figure 13 Ornanogram of composite fibres (Saba, Tahir, and Jawaid 2014)

By way of example of the advantages of natural fibres, there are presented next some reasons that make them environmentally superior to the glass fibre composites, (Joshi et al. 2004):

- Natural fibre production has lower environmental impacts compared to glass fibre production;
- Natural fibre composites have higher fibre content for equivalent performance, reducing more polluting base polymer content;
- The light-weight natural fibre composites improve fuel efficiency and reduce emissions in the use phase of the component, especially in auto applications;
- End of life incineration of natural fibres results in recovered energy and carbon credits.

For those and other reasons, natural fibres are growing on production. Table 2 shows different natural fibres used in composites, these ones especially used in concrete, which were produced annually globally.

Table 2 Estimated 2013global volume production of different natural fibres (EchoTech Alliance, 2014)

Fibre Source	Production per year (10^3 ton)	Main producer countries
Jute	2.5	India, Bangladesh
Kenaf	0.45	China, India, Thailand
Sisal	0.30	Brazil, China, Tanzanya, Kenya
Hemp	0.10	China
Ramie	0.15	China
Abaca	0.10	Philippines, Equator
Cotton	25	China, USA, India, Pakistan
Silk	0.10	China, India

Disadvantages

The main disadvantages of natural fibres in respective composites are:

- The poor compatibility between the fibre and the matrix and their relatively high moisture absorption - what some surface treatment could improve the problem;
- Thermal and mechanical degradation during processing - what make them undesirable for certain applications;
- Poor wettability.

However, broad varieties of fibres with different thermal and mechanical properties are abundantly synthesized in nature and are available for the development of high-performance composites.

It is required, to be utilised properly, to know some of that properties of those fibres. When determining the properties, it is crucial to understand that natural products have properties that are strongly influenced by their growing environment. Conditions such as temperature, humidity, the composition of the soil and the air all have effect at the height of the plant, strength of its fibres, density, etc. Also where the plant fibres are sourced, the way the plants are harvested, collected and processing methods result in a variation of properties, (K. van Rijswijk, W.D. Brouwer, and Beukers 2003).

For comparison between the synthetic and natural fibres, there is shown on Table 3 some properties.

Table 3 Mechanical properties of some common natural and synthetic fibres (adapted from Becerra et al. 2014)

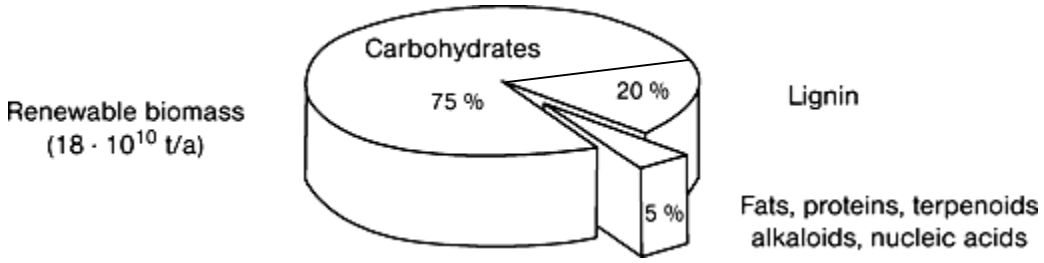
Natural fibres	Density (g/c^3)	Tensile Modulus (GPa)	Tensile Strength (GPa)	Failure Strain (%)
Flax	1.52	100	0.84	2.0
Hemp	1.52	7	0.92	0.92
Kenaf	1.52	53	0.93	1.6
Sisal	1.52	38	0.88	2.7
Wood	~1	10 – 80	~1.5	1 – 3
Jute	1.52	60	0.85	2.0
Synthetic fibres				
Glass	2.5	72	2.5	2.5
Carbon	1.9	380	2.0	1 – 2
Aramid	1.4	125	2.8	2 – 4
Metals				
Aluminium	2.8	73	0.47	10
Steel	7.8	200	0.40	30

Biomass

Sources of bioenergy called "biomass" include agricultural and forestry residues, municipal solid wastes, industrial wastes, and terrestrial and aquatic crops grown solely for energy purposes. If referred to plant-based materials, it is called lignocellulosic biomass. Therefore, the natural fibres also make part of the terrestrial biomass. This biomass constitutes a cheap and easily accessible source of raw materials for the bio-based industry such as biomaterials, biochemicals and biofuels. The 30€/ton of biomass are far incomparable with synthetic material prices, being an incentive for forest cleaning to the collecting of this raw material.

The biomass collection can improve forest products and by-products valorisation as a tool to reduce fire risk. While responding to specific environmental problems, by being included on new value added products, it creates a new source of income. The biomass, particularly the forest waste on lying on forests and available at the pulp mills wood yard can be utilized for the transition from fossil to renewable raw materials.

However, when compared to fossil resources, it is an exceedingly complex array of low and high molecular mass products, exemplified by sugars, hydroxy and amino acids, lipids, and biopolymers such as cellulose, hemicelluloses, chitin, starch, lignin, and proteins. According to Lichtenthaler, 2010, the most significant class of organic compounds (produced in terms of volume) are the carbohydrates (from which cellulose makes part), representing roughly 75% of the annually renewable biomass of about 180×10^9 t, as represented on Graphic 5. Of these, only a minor fraction (ca. 4%) is availed by man while the rest decays along natural pathways.



Graphic 5 Distribution of types of natural products in biomass (Lichtenthaler, 2010)

Thus, carbohydrates, a single class of natural products — apart from their traditional uses for food, lumber, paper, and heat — are the major biofeedstocks from which to develop industrially and economically viable organic chemicals that are to replace those derived from petrochemical sources.

Fibres composition

Plant fibres comprise mostly lignin, cellulose, and hemicellulose. The composition can vary between hardwood and softwood, as shown on Table 4, and from fibre to fibre, as shown on Table 5.

Table 4 Wood components (Sjostron, E., 1993; Rowell, R., 1993)

	Hardwood (%)	**Softwood (%)**
Cellulose	41 – 51	33 – 42
Lignin	21.5 - 31	27 – 32
Hemicellulose	20.0 – 34.5	26 – 33
Ash	0.2 – 1	0.1 – 0.5

Table 5 Chemical composition of common lignocellulosic fibres (Dai and Fan 2014)

Fiber	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Extractives (%)	Ash Content (%)	Water Soluble (%)
Cotton	82.7	5.7	—	6.3	—	1.0
Jute	64.4	12	11.8	0.7	—	1.1
Flax	64.1	16.7	2.0	1.5–3.3	—	3.9
Ramie	68.6	13.1	0.6	1.9–2.2	—	5.5
Sisal	65.8	12.0	9.9	0.8–0.11	—	1.2
Oil palm EFB	65	—	19.0	—	2.0	—
Oil palm frond	56.0	27.5	20.5	4.4	2.4	—
Abaca	56–63	20–25	7–9	3	—	1.4
Hemp	74.4	17.9	3.7	0.9–1.7	—	—
Kenaf	53.4	33.9	21.2	—	4.0	—
Coir	32–43	0.15–0.25	40–45	—	—	—
Banana	60–65	19	5–10	4.6	—	—
PALF	81.5	—	12.7	—	—	—
Sun hemp	41–48	8.3–13.0	22.7	—	—	—
Bamboo	73.9	12.5	10.2	3.2	—	—
Hardwood	31–64	25–40	14–34	0.1–7.7	<1	—
Softwood	30–60	20–30	21–37	0.2–8.5	<1	—

2.2.5. Natural Matrix

While the fibrous reinforcement provides stiffness and strength and carries most of the structural loads, the composites' shape, surface appearance, environmental tolerance and overall durability are dominated by the matrix.

Commodity plastics dominate the polymer market with 80% of consuming materials based on non-renewable petroleum resources. Governments, companies and scientists are driven to find alternative matrices to the conventional petroleum based matrix, through public awareness of the environment, climate change and limited fossil fuel resources. Therefore, bio-based plastics, which consist of renewable resources, have experienced a renaissance in the past few decades and as a result, new bio-composites with bio matrices have been developed - considering a bio matrix as one that is biodegradable, either natural or oil based.

Degradable plastics

Importance of biodegradable polymers/composites assumes significance due to the problem of the solid waste generated by plastic materials after their final use. Many definitions of biodegradable polymers/composites are proposed of which most acceptable one seems to be as follows: "*Those materials obtained from nature or by synthetic route, whose chemical bonds are cleaved at least in one step by enzymes from the biosphere, with appropriate pH and temperature conditions and total processing time for completion"*(Bogoeva-Gaceva et al. 2007).

Natural matrices are then generally formed by biodegradable polymers are widely accepted with the division into three main categories in literature, depending on whether the polymerization is biological or synthetic, (Bogoeva-Gaceva et al. 2007):

I. Biosynthetic – natural polymers produced from natural sources (i.e. cellulose or starch) or produced in the nature by fermentation of sugars or lipids (i.e. polyhydrohyalkanoates) they are biodegradable and have similar mechanical and thermal properties to polyolefins like PP and PE;

II. Semi-biosynthetic – polymers in which the monomer unit is produced naturally or by a fermentation process, and then the polymerization procedure is a classical synthetic procedure. A representative of the semi-biosynthetic polymers is polylactic acid (PLA);

III. Chemosynthetic polymers – belong to the family of polyesters. Polycaprolactones (PCL) is one of the representatives of this class of polymers.

The polymeric matrices are the most studied in the production of composite materials with natural fibres serving as reinforcement. On Figure 14 there is an organogram showing the principal polymeric matrices used for bio-composites manufacturing (mainly the manufacturing using natural fibres as reinforcement) and Figure 15 shows biodegradable polymers that constitute bio-degradable matrices with more classification detail.

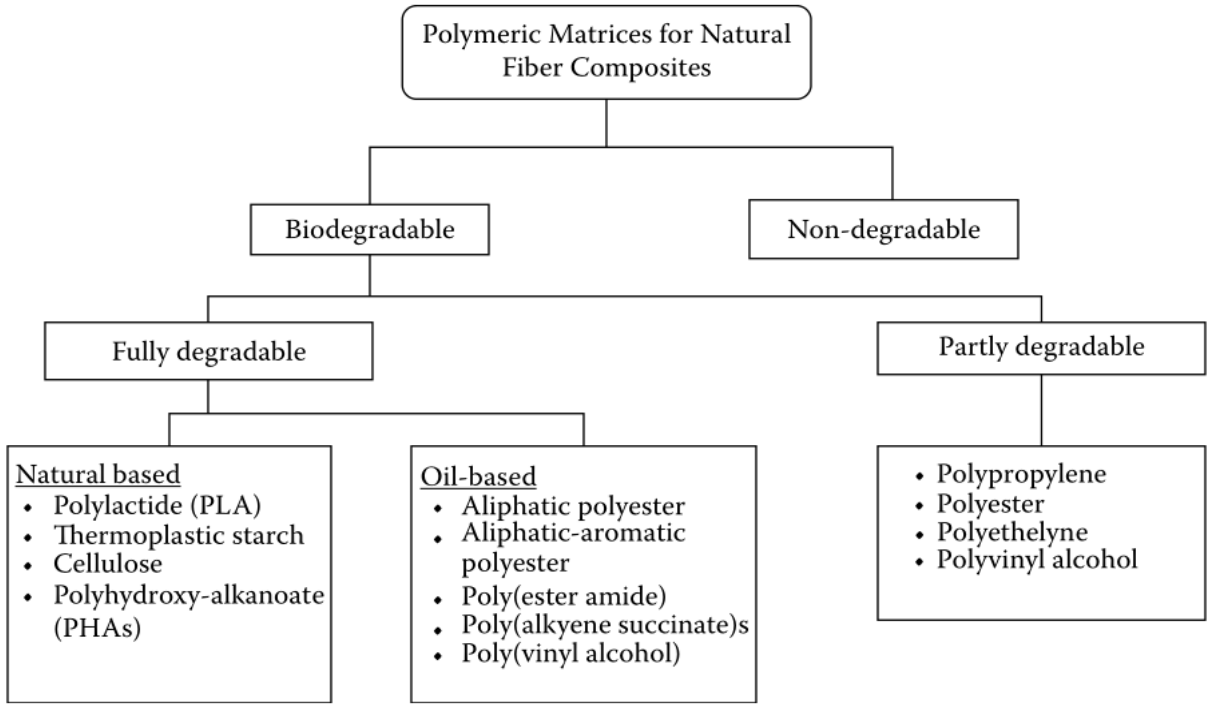


Figure 14 Organogram of polymeric matrices for biocomposites (Taylor & Francis Groups 2016)

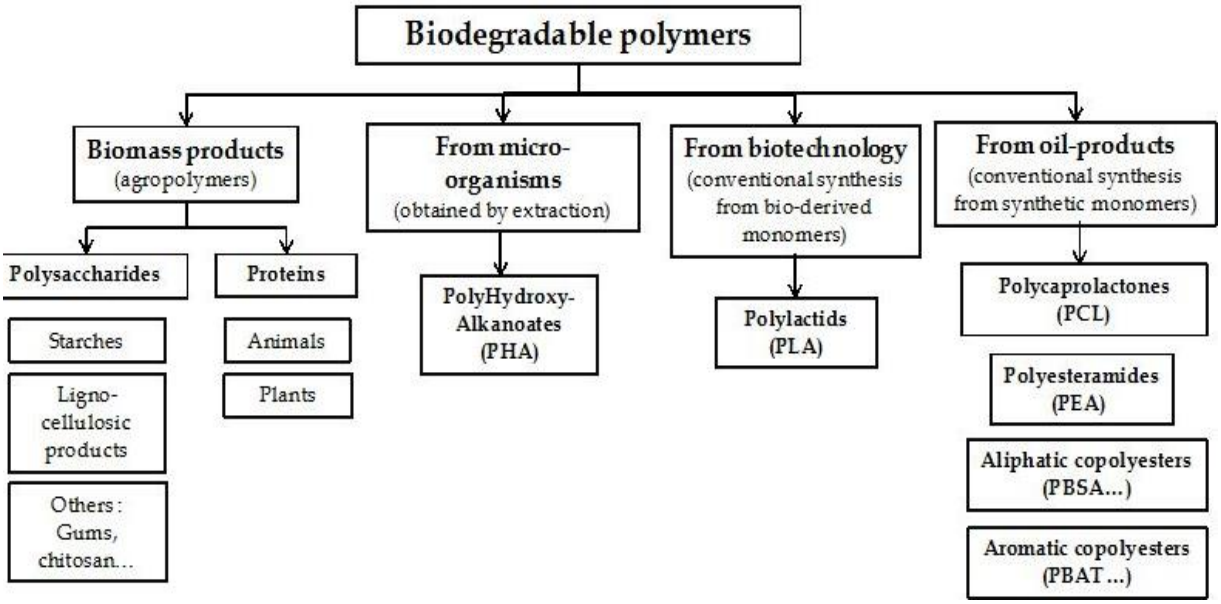


Figure 15 Classification of biodegradable polymers, in Averous (2004)

From those, the polymers most commonly used as bio-matrices together with natural fibres as reinforcements are:

- Starch - a naturally occurring polymer;
- Polylactic acid (PLA) - using raw materials for its manufacturing of lactic acid, the monomer is obtained from the fermentation of sugar;
- Polyhydroxy-alkanoate (PHA) - produced from vegetable oils;

Other bio-based polymers that have been studied more as matrices are:

- Soy-based resin - from soy oil;
- Polycaprolactone (PCL);
- Polybutylene (PBS), to a lesser extent than the first ones.

Bioplastics

FKuR Kunststoff GmbH is a company that has got the slogan "Plastics – made by nature" as it has developed a wide range of biodegradable plastics primarily made from natural resource materials. FKuR has developed a diverse range of different grades for extrusion, thermoforming and injection moulding. Dependent upon the grade, FKuR products guarantee a long service life or are biodegradable and break down into naturally occurring, harmless base materials. Some examples are on Figure 16.

BioFlex	**Biograde**	**Fibrolon**	**Terralene**
Biodegradable plastics for extrusion	Biodegradable plastics for injection moulding	Natural fibre reinforced plastics	Biobased Compounds based on Green PE

Figure 16 FKuR biomaterials (FKuR biobased)

Bioplastics possess a tremendous market potential and have higher market growth rates compared to standard plastics. Europe is the largest consumer of bioplastics, accounted for more than one third of total global bioplastics consumption in 2013. Thus, this region is the most important sales market, followed by North America, Asia-Pacific, and the rest of the world, according to Ceresana[8].

Drop-In bioplastics

"Braskem is the world leader in the field of biopolymers because of Green PE, first produced on a commercial scale in September 2010". Green polyethylene is product that is obtained from sugar cane ethanol, a renewable source. Green PE also maintains similar characteristics to the traditional PE. Although this plastic has a biological source, it is not biodegradable. Non-biodegradable bioplastics, which are also known as "Drop In" bioplastics, include products such as bio-PE or green-PE, or bio-PET, that possess properties similar to their equivalents made from fossil resources. The packaging industry is the main market. The major advantage of cups, bottles, plates, bags, and sacks made from biodegradable bioplastics is the fact that they can be disposed of along with leftover food.

2.2.6. Case Study

Swedish researchers from KTH have recently produced (on February of 2016) a car which roof and batteries are made from wood-based carbon fibre. Although the prototype is still on a small scale, it is a great step towards new lightweight materials made from forest sources. In this case, wood lignin, a by-product from paper pulp production, was used. Göran Lindbergh, Professor of Chemical Engineering at KTH, said that the use of this material as an electrode material came from previous research he did with Innventia[9]. The prototype is shown on Figure 17.

[8] Ceresana is a leading international market research and consultancy company for the industrial sector.

[9] Innventia is a world-leading research institute that works with innovations based on forest raw materials, relating to pulp, paper, graphic media, packaging and bio refining.

Figure 17 KTH car lignin-based prototype (KTH, research news, 2016)

2.3. Eucalyptus tree

History

Late in the XVIII century, L'Heritier De Brutelle, a French botanist, applied the name Eucalyptus to a tree with its origin in Australia. Mostly because the wood was difficult to saw and season at that time, there was little appreciation of the potential of eucalypts to become a good source for forest-derived products (Turnbull 1999).

The major planting of the eucalypts, outside its Australian native environment, the Malaysian region, and the Philippines, started in 1904 in Brazil (Dessie and Erkossa 2011). There, in the early 1900s, eucalypts were seen as the answer to the scarcity of hardwood timber in the United States and were heavily promoted and extensively planted in California. Planting rates of the eucalypts approximately doubled each decade until the end of the 80's (Turnbull 1999). Planting was curtailed, though, when the difficulties in sawing eucalypt wood became evident.

The reasons for replanting eucalypts have been changing significantly over time, and the end uses to which the species have been applied are diverse: sawn timber, mine props, paper and paper pulp, fibreboard, poles, firewood, charcoal, essential oils, honey and shade, products and materials. Remarkably, nowadays the eucalypts have become one of the most widely planted genuses in the world.

It is among the most preferred trees because it grows fast and has a good survival capacity in marginal environments. In Portugal is the most preferred one by cellulose industry, due to the facts referred before plus the good quality of wood.

There are nine species that seem to be the most used between the large number of the eucalyptus that have been planted throughout the tropics and sub-tropics: *E. camaldulensis, E. globulus, E. g randis, E. maculata, E. pa niculata, E. robusta, E. saligna, E. urophylla, and E. viminalis*" (Dessie and Erkossa 2011), being the specie *E. regnans* considered as the tallest tree on Earth, with 140 meters.

Both arguments with which the success and widespread cultivation of eucalypts is related and the ones against the plantation of eucalypts are referred on Table 6, but its negative environmental impacts have often been controversial.

Both the total area of plantations and the rate of increase grew considerably in the following years, as illustrated in Table 7, which was based on a sample of 61 countries with significant areas of plantations.

Table 6 Advantages and disadvantages or dangers of eucalyptus plantation (adapted from (Peter Holland 2009; Liu and Li 8152; João Lé 2012)

Advantages	Disadvantages
Fast growing tree	Suppresses undergrowth
Exceptional drought-hardiness	Drains water resources
Requires minimum care	Enhances soil erosion
Grows in wide ecological zones and poor environments	Depletes soil nutrients
Coppices after harvest	Introduces allelopathic[10] effects
Resists environmental stress and diseases	Fire propagation
The seeds are easy to collect, store and no pre sowing treatment is required	Falling of branches
Easily cultivated and managed	
Wood of high and calorific value	
Good economic returns	
Good properties to human health	

Table 7 Plantation area 1990-2005, by function 1 000 ha (FAO, 2006)

Region (countries sampled)	1990		2000		2005	
	Productive	Protective	Productive	Protective	Productive	Protective
Africa (58)	10 163	2 083	10 581	2 283	10 876	2 462
Asia (47)	28 925	17 666	36 206	19 459	44 414	20474
Europe (47)	17 942	4 588	20 997	5 591	21 651	6 027
North & Central America (37)	10 595	187	16 711	1 227	17 653	1 190
Oceania (24)	2 447	1	3 477	14	3 833	32
South America (15)	9 094	39	11 383	54	12 132	57
World	79 165	24 562	99 356	28 628	110 560	30 529
	103 727		127 984		140819	
Rate of change			90-00, 1.7%/year		00-05, 1.9%/year	

Eucalyptus fibres

As said on chapter 2.2.4, while the man-made fibres as the synthetic fibres are based on fossil fuels as crude-oil, the eucalyptus fibres coming from a tree are based on the most naturally occurring organic polymer: cellulose.

The fibre length of Eucalyptus is relatively short and uniform, with low coarseness compared with other hardwoods commonly used as pulpwood. The fibers are slender, yet relatively thick walled.

In the study (Clarke et. al 2008) the fibre properties of macerated wood samples from a range of *Eucalyptus* species used commercially in South Africa were compared with those of North

[10] Allelopathy is the inhibitory effect by one specie to another through a chemical release

American hardwoods such as birch, maple and aspen. In that comparison, the *Eucalyptus* species were found to have short and thin fibres, as seen on Table 8.

Table 8 Average width and length of commercial South Africa *Eucalyptus* and North America hardwoods (Clarke et. al 2008)

Tree	Fibre length	Fibre width
South Africa *Eucalyptus*	0.6 to 0.8 mm	15 to 17 μm
North American hardwoods	0.6 to 1.4 mm	17 to 30 μm

The *Eucalyptus* fibre is therefore reasonably fragile.

Another investigation of fibre lengths but this time through the variation during the course of the ring from one tree of *Eucalyptus regnans*, 50 years old and, 52m high, had found that the fibres of the last-formed late wood were longer than those of the early wood. "The fibre length in the late wood varied throughout the tree from 0.6 mm in the ring next to the pith at all levels, to 1.35 mm the outermost rings at the 15m level. At all levels there was a rapid increase in length of both early and late wood fibres from the centre of the tree outwards for approximately 10 growth rings, after which a more or less constant value was reached in each case.", (BISSET and DADSWELL 2013).

Eucalyptus to paper

The physical and chemical attributes of the wood from *Eucalyptus* contribute to its popularity in the pulp and paper-making industries. The particular combination of dimensions from *Eucalyptus* fibres, produces low fibre coarseness, which is a highly desirable attribute for products such as coated and uncoated papers. However, those sizes make it particularly vulnerable to damage during the pulp and bleaching processes. Fibre damage occurs throughout the pulp process but is most severe in the mechanical sections such as digester blowing, high shear mixers, medium- and high-consistency pumps as well as low-consistency refining.

Even though, bleached *Eucalyptus* pulp is used extensively and globally in paper-making in such diverse products as tissue, packaging, and printing papers, from high quality. Chemical cellulose products such as viscose, acetate and microcrystalline cellulose are also made from them.

Kraft process[11]

The Kraft process is also known as Kraft pulping or sulphate process. The objective of pulping is to separate wood into individual fibres. Kraft pulping converts wood into wood pulp, also called cellulosic pulp, as it consists of almost pure cellulose fibres, the main component of the outsider of wood and therefore, the main component of paper.

This process involves several steps, both chemical and mechanical such as heat and pressure. However, essentially, it entails treatment of wood chips with the so called white liquor - a hot mixture of water, sodium hydroxide, and sodium sulphide – that removes the bonds between the lignin and hemicellulose and cellulose. This is exemplified on Figure 18. The bad points of Kraft plants are that they can release smelly products and substantial liquid wastes.

[11] Origin on a German word - Kraft - meaning "strength", due to the strength of the kraft paper produced using this process. Both capitalized and lowercase spelling "Kraft process" and "kraft process" appear in the literature, but "Kraft" is most commonly used in the pulp and paper industry.

The Kraft process is a cyclical, self-sustaining process. The black liquor formed as a result of the process is a combination of the removed lignin, water, and chemicals used in the extraction process. This black liquor is concentrated through evaporation and then burned in order to generate high-pressure steam for the mill processes used to make paper. The inorganic portion of the black liquor then regenerates the sodium sulphide and sodium hydroxide that is used for pulping.

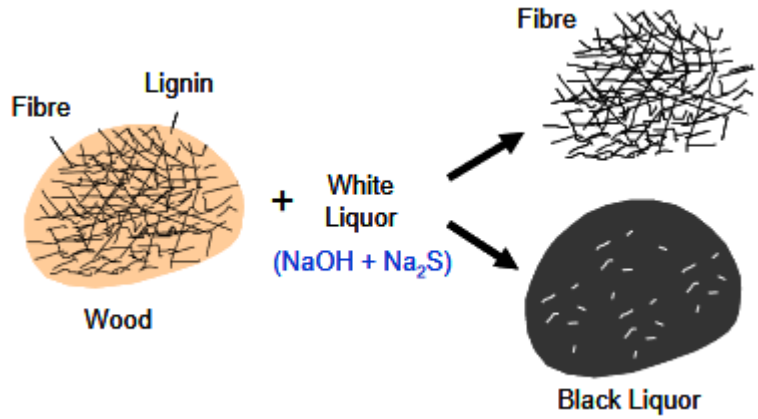


Figure 18 Kraft pulping process (Tran and Vakkilainnen 2016)

About 130 million tons/year of Kraft pulp are produced globally. The high strength of Kraft pulp, the ability of the process to handle almost all species of softwood and hardwood, and the favourable economics due to high chemical recovery efficiency (about 97%) give the Kraft process an advantage over other pulping processes (Tran and Vakkilainnen, 2016).

Bark of Eucalyptus

For the growing of the tree, the xylem has the basic function of transporting the water and nutrients from roots to shoot and leaves. The bark of tree is a very important protection for the xylem against the weather and threats of the environment, insects, fungus and others. It means that the bark is stronger and has a harder degradability than the xylem.

The amount of fibres present in the bark is less than in the trunk because bark does not have the function of ensuring the stability and structural reinforcement of the tree. The constitution of the interior of a trunk is shown on Figure 19.

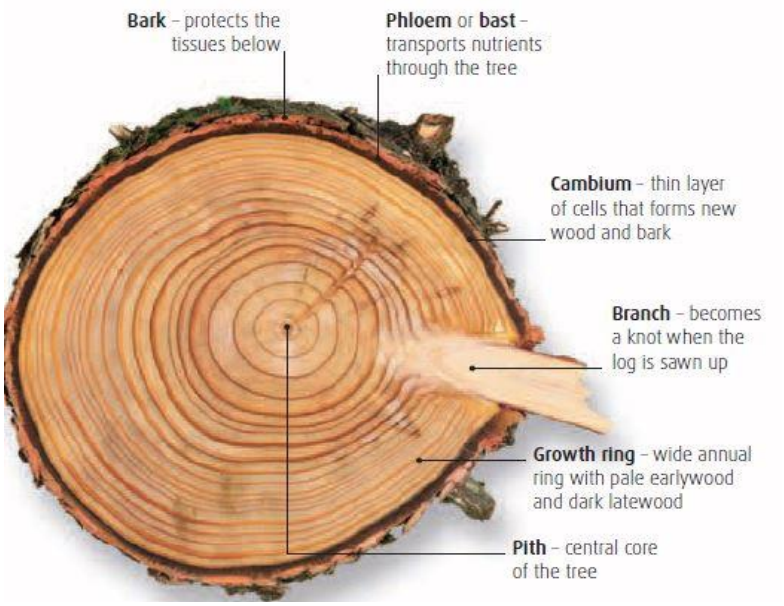


Figure 19 Diagram of the constitution of eucalyptus trunk (Wood Chip Marine Lumber)

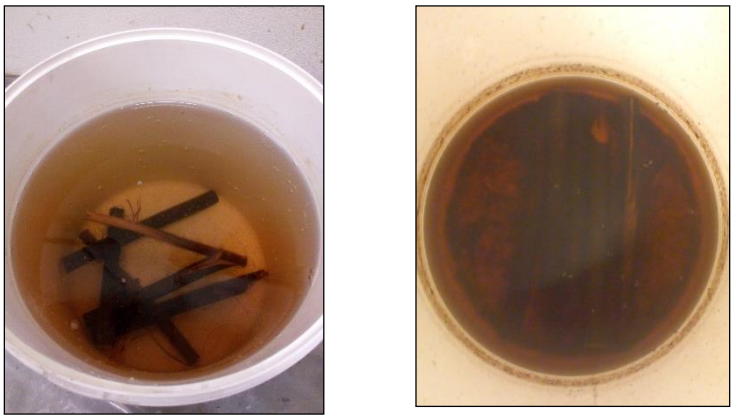
The bark of *E.globulus* tree is sleek and once it is dead, it liberates itself from the trunk. These plainer barks are the easiest to remove on harvesting operations. When the bark falls on the floor together with leaves and branches, as seen on Figure 20, they feed the cycle of nutrients between the floor and the plant.

Figure 20 Bark of *E.globulus* (Mount Sutro Open Space Reserve)

One eucalyptus tree for commercialization has 10% to 18% of its total trunk volume as being bark, but the cloned trees genetically modified for high volumetric increase can have 9% to 12% of bark, in volume, (Foelfel 2015). The first ones are considered of less value for paper industry, because of lack in fibres and surplus in phenolic components. It means that the less amount of bark in terms of weight and volume, the better is the tree considered for commercialization. Cellulose companies often peel the Eucalyptus trunks just after the harvesting of the trees, and leave the barks on the field, as seen on Figure 21.

Figure 21 Harvesting operations with bark peeling (Illingworth Ingham M/Cr), (AFM-Forest 2016)

A way of measuring the density of the bark of the of Eucalyptus trees is soaking the material in water for some days and obtaining the so called green or saturated volume. But the bark as great solubility in water, cold or hot, so the loss of weight is considerable, causing a brown colouring on the water due the loss of the organic matter, as seen on Figure 22 on an experience of soaking eucalyptus poles to better understanding.

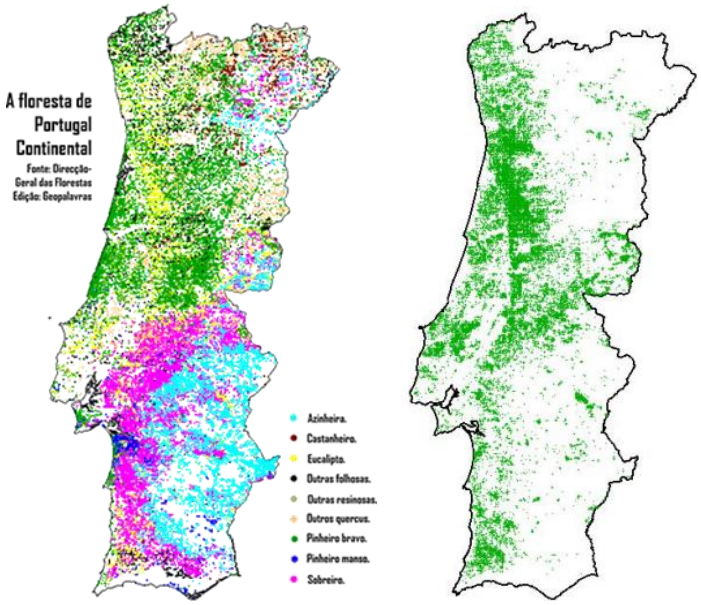


(a) (b)

Figure 22 Soaked eucalyptus poles on water: (a) 7 days; (b) 60 days

2.3.1. Panorama in Portugal

The forest of Portugal corresponds to about 35% of Portugal territory, excluding the islands. It corresponds to more than 3 000 000ha (about 3 154 800ha). The eucalypt trees dominate the forest occupation of the country, on about 26%, correspondent to 812 000 ha, (~52m^3/ha). At the centre region of the country is localized 50% of it. This can be seen on Figure 23, and also the distribution of *Eucalyptus.*



(a) (b)

Figure 23 Distribution of the main species in Portugal (a); Distribution of *Eucalyptus* in Portugal (b) (CNF, 2013)

The continuous growing area occupied with eucalypts is directly related to the cellulose industry as seen on Figure 24.

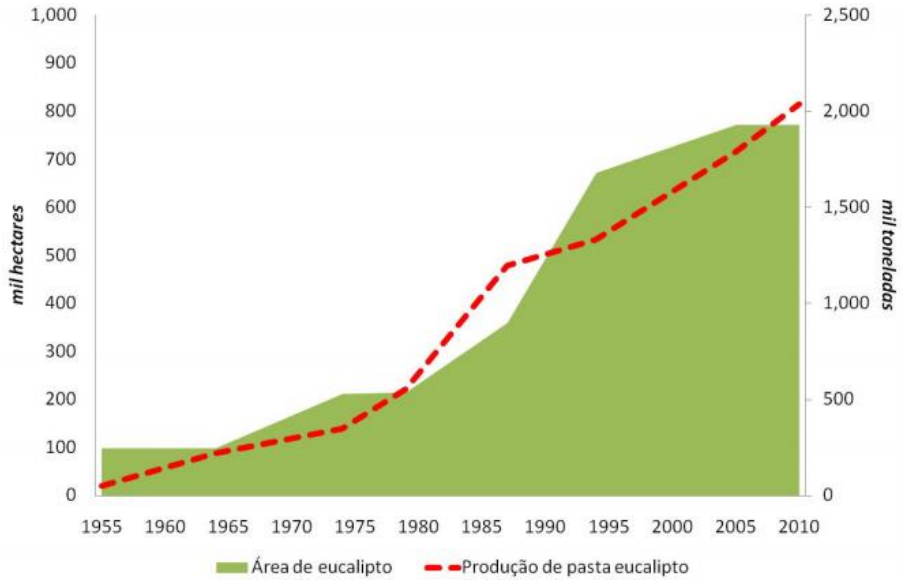


Figure 24 Growth of Eucalyptus' area during time (CNF, 2013)

The economic relevance of cellulose industry in Portugal is shown on Figure 25, representing approximately 10% of the Portuguese' goods exports (~42M€).

Figure 25 Contribution of the cellulose industry on Portuguese exports

Cellulose industry

The cellulose industry harvest wood to produce their pulp that will be ultimately processed in paper. Though it started with pine, nowadays the wood serving this industry is *Eucalyptus globulus*. This tree is harvested after an average growing of 8 to 10 years. After two to three harvests, the principal trunk starts growing surrounded by some little new trunks, the poles, around the main one, as it shown on Figure 26. Once the poles are cut they are considered forest waste. Sometimes they are collected together with branches, leaves and other forest residues, for the production of electrical and thermal energy, mainly on their own mills. Other times, both because the soils need to be fed from nutrients, minerals and other components or because of some impossibility to transport those residues, they are left on the field for some time.

Figure 26 Eucalyptus tree with new poles growing

Associated to the dry summer of Portugal there is an ease in the ignition according to the dryness of the vegetation, being the biomass residues boosters to fires. Therefore, forest wastes present on forests and available at the pulp mills wood yard constitute a cheap and easily accessible source of raw materials for the bio-based industry such as biomaterials, biochemicals and biofuels. The 30€/ton of biomass are far incomparable with synthetic materials prices, being an

incentive for forest cleaning. The biomass collection can improve forest products and by-products valorisation as a tool to reduce fire risk. While responding to specific environmental problems, it creates a new source of income.

With the focus on natural renewable vegetable fibres from forest waste and recycled materials, the objective of this project is the characterization of a bio-eco-composite.

2.4. Companies dealing with natural fiber composite

Context

Day by day, the demand for green alternatives is getting stronger. In December 2015 the president of the USA, Barack Obama, officially signed a law prohibiting from July 2017 on, the manufacturing of products containing plastic microbeads, like exfoliating skin care products, toothpaste and shampoo; and the introduction or delivery introduction into interstate commerce is prohibited from July 2018 on. One tube of exfoliating facewash can contain more than 350,000 microbeads, which means microbeads are a small part of the more than eight million metric tons of plastic that get into the world's oceans each year. Yet, once in the environment, plastic microbeads may be eaten by every ocean's animals, and then transfer their concentrate toxins such as pesticides and flame retardants, to the animal's tissues, contributing to a pollutant life cycle.

JRS, J. RETTENMAIER & SÖHNE Group

Between many other applications JRS offers cellulose as an alternative to plastic microbeads. They have dedicated to research, development and processing of high quality organic fibres derived from vegetable raw materials for industrial purposes. These include products made of cellulose, cereal, fruit fibres or woodfibers, processed into microfibers, compacts, granules, mixtures or special dosage forms as needed.

Direct comparison shows the advantages between PE and biomaterials is shown on Figure 27.

The main things:	INCI: Cellulose or INCI: Microcrystalline Cellulose	INCI: Polyethylene
White color	Yes	Yes
Fast and easily biodegradable	Yes	No
Made from renewable resources	Yes	No
ECOCERT / COSMOS / NPA certified	Yes	No
Safe and edible	Yes	No
Safe food for animals	Yes	No
Ecofriendly	Yes	No
Consumer likes it	Yes	No
Source 	Yes	No

Figure 27 Differences between cellulose materials and polyethylene (J. RETTENMAIER & SÖHNE, 2016)

Other actual companies that produce composites based on cellulose or based on ecologic solutions, are listed next:

- Mondi Group – Mondi developed FIBROMER® which is a reinforced polymer with cellulose fibre; in a new compounding process that homogeneously mixes Kraft pulp (a renewable resource) with a polymer granule. The result is a new composite that offers clear advantages for injection moulders and original equipment manufacturers compared with other composites used in manufacturing, such as those containing short glass fibres, talcum-filled materials as well as other natural fibres, (mondigroup, 2016)

- Biofore company – Biofore company developed UPM Formi which is a cellulose fibre reinforced plastic composite. As a result, cellulose fibres significantly increase the stiffness and strength of polypropylene, creating value from renewable and recyclable materials, (UPM, 2016);

- Tinta Marta – Indonesian's company makes plastic from manioc. This biodegradable plastic is called Ecoplastic. It can be degraded in weeks when buried amongst active microbes or insects, depending on microbial activity level. Brands like Zara and GAP already use it on their standard shopping plastic bags (ecoplas, 2016)

- Reform Studio – This Company turns recycled plastic bags into designer furniture with a traditional Egyptian weaving process. Plastex is a new eco-friendly material made by weaving of discarded plastic bags. This is a rather ingenious solution to our plastic bag epidemic, in which the bags become the feedstock for a traditional, yet disappearing, industry in Egypt – handweaving (reformstudio, 2016)

- Sodra – Durapulp is a bio-composite material that consists of completely renewable and bio-degradable components. Its environmental credentials are undeniable, but Durapulp boasts several other unique features as well, (Sodra, 2016)

2.5. Manufacturing processes of composites

There are numerous methods for fabricating composites. Some of those methods had already existed before and were just adapted, like injection moulding, but other have been developed to come across specific designs or manufacturing challenges. The selection of a method will depend on various variables including the materials, the design, and end-use or application. Following are some brief description of manufacture processes of composite materials.

Hand layup

Hand layup, the most basic manufacturing process for thermoset composites, typically consists of laying, by hand, dry fabric layers, "plies," or prepreg plies, onto a tool to form a laminate stack. After layup is complete, a resin is applied to the dry plies. Other technique is wet layup on which each ply is coated with resin and compacted after it is placed.

But the demand for faster production rates has pushed the industry to replace hand layup with alternative fabrication processes and has encouraged fabricators to automate those processes wherever possible. (Gardner Business Media 2014).

Resin transfer moulding (RTM)

Resin transfer moulding is a simple liquid moulding process. It begins with a two-part, matched and closed mould, made of metal or composite material. Dry reinforcement (typically a

preform) is placed into the mould and it is closed. Resin and catalyst are metered and mixed in dispensing equipment and is injected into de mould at low or moderate pressure, following predesigned paths through the preform. The resins used in RTM are less expensive than prepreg material and can be stored at room temperature. The process can produce thick parts, eliminating most post-fabrication work; dimensionally accurate complex parts with good surface detail and delivers a smooth finish on all exposed surfaces. RTM produces parts without an autoclave though a part for a high-temperature application usually undergoes postcure.

Other techniques growing in popularity are: light RTM, high-pressure RTM (HP-RTM) and vacuum-assisted RTM (VARTM). In light RTM, low injection pressure under vacuum allow the use of less-expensive, lightweight two-part moulds or a very lightweight, flexible upper mould. High-pressure RTM (HP-RTM), typically designed as a completely automated system that shows promise for high production once it has the ability to rapidly fill a mould loaded with a preform with a very fast curing resin. Vacuum-assisted resin transfer moulding (VARTM) represents the fastest-growing new moulding technology, on which resin is drawn into a preform through the use of a vacuum only, rather than pumped in under pressure and high heat.

Pultrusion

Pultrusion has been used for a long time, like RTM. It is a continuous process where the reinforcing fibre is generally a roving, a tow or a continuous mat. This fibre is typically pulled through a heated resin bath and then it passes through one or more forming guides or bushings to form into specific shapes. Then, the material moves through a heated die, where it takes its net shape and cures. After cooling, the resulting profile can be cut into the desired length. Pultrusion yields smooth finished parts that typically do not require post processing.

Injection moulding

Injection moulding is a fast, high-volume, low-pressure, closed process using, most commonly, filled thermoplastics. A ram or screw plunger forces a metered shot of material through a heated barrel and injects it into a closed heated mould. After cure and ejection, parts need only minimal finishing. Injection speeds are typically one to five seconds, and as many as 2,000 small parts can be produced per hour in some multiple-cavity moulds.

Injection moulding is the most common process for the production of plastic components worldwide. Some examples can be found within the automotive, construction, furniture and toy industries or applications for electrical appliances and household goods.

Compression moulding

Compression moulding demands expensive but durable metal dies for its high-volume thermoset moulding process, being an appropriate choice when production quantities exceed 10,000 parts. As many as 200,000 parts can be turned out on a set of forged steel dies, using sheet molding compound (SMC), a composite sheet material made by sandwiching chopped fiberglass between two layers of thick resin paste. To form the sheet, the resin paste transfers from a metering device onto a moving film carrier. Chopped glass fibres drop onto the paste, and a second film carrier places another layer of resin on top of the glass. Rollers compact the sheet to saturate the glass with resin and squeeze out entrapped air. The resin paste initially is the consistency of molasses); over the next three to five days, its viscosity increases and the sheet becomes leather-like ideal for handling."

Filament winding

Filament winding is a continuous fabrication method that can be automatized with relatively low material costs. A mandrel is suspended horizontally between end supports, while the "head" — the fibre application instrument — moves back and forth along the length of a rotating mandrel, placing fibre onto the tool in a predetermined configuration. Computer-controlled filament-winding machines are available.

In most thermoset applications, called wet winding, the filament winding apparatus passes the fibre material through a resin "bath" just before the material touches the mandrel, or uses towpreg, a continuous pre-impregnated fibre, which eliminates the need for an onsite resin bath. Fibre can also be wounded without resin (dry winding). The dry shape is then used as a preform in another moulding process, such as RTM. In thermoplastics winding the material is in prepreg form, so a resin bath is not needed. Material is heated as it is wound onto the mandrel.

2.6. Composite materials recycling

The waste management and environmental legislations call for all engineering materials, as of end-of-life products, to be suitable to recover and properly recycled. Currently, composites have not been properly recycled mostly due to their heterogeneity hybrid structure and thermoset base. There is still work to be done on development of much more efficient separation technologies and creation of better recyclable composites. There are technologies still being commercialized mostly focusing on reinforcement fibres, as seen on Figure 28, mechanical recycling, thermal recycling and chemical recycling that could be applied both to materials and energy recover. But lack of adequate markets, high recycling cost, or lower quality of the recyclates are the major commercialization barriers. However, joint efforts from product-design, manufacturing, and end-of-life management, new separation and recycling technologies for the composite materials will be available and more easily recyclable composite materials will be developed in the future. Recycling will ultimately lead to resource saving (Yang et al. 2012).

2.6.1. Recycling Processes

There are three main processes of recycling plastics, both thermoplastics and thermosetting: mechanical, chemical and thermal, as shown on Figure 28.

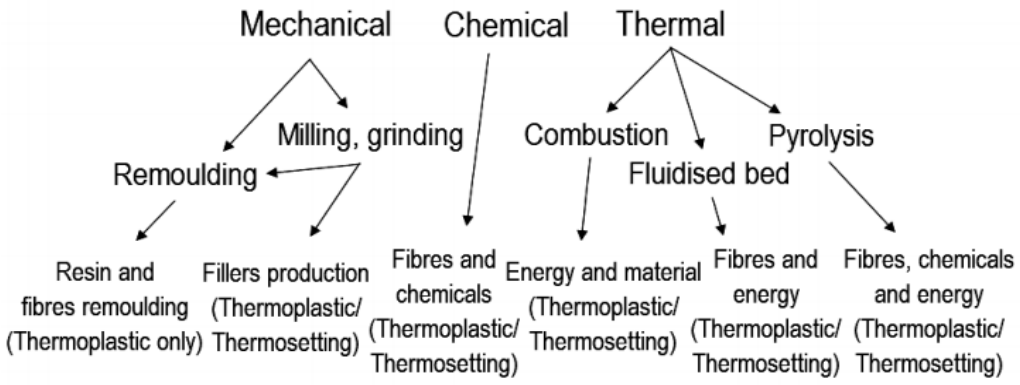


Figure 28 Recycling processes for thermoplastic and thermosetting composites (Otheguy et al. 2009)

On Table 9 there are shown the definitions according to the norms ASTM D5033, ISO 15270 equivalent terms off which they are also known, and a brief description.

Table 9 Terminology used in different types of plastics recycling, (Hopewell et. al 2009), (Raj 2015).

ASTM D5033 definitions	Equivalent ISO 15270 definitions	Equivalent terms	Description
Primary recycling	**Mechanical recycling**	Closed-loop recycling	Reuse as new product with similar properties
Secondary recycling	**Mechanical recycling**	Downgrading	Reuse as new product with less demanding properties
Tertiary recycling	**Chemical recycling**	Feedstock recycling	Converting waste into basic chemicals
Quaternary recycling	**Energy recovery**	Valorisation	Waste incineration to recover energy content as heat

2.6.2. Primary recycling

Primary recycling means that the recyclable material/product is recovered and reused without being changed and usually for the very same purpose. It involves processing of waste into a product with characteristics similar to those of original product. In a first way, primary recycling can be defined as second hand use. Some examples are reusing yourself, donating or selling.

2.6.3. Secondary recycling

Secondary recycling involves some sort of modification of the material/product, and without the use of chemical processes. It means that the material/product is reused in some other way without reprocessing, (Recycling Consortium 2014), but it was turned into a product that have characteristics dissimilar from those of the original. Examples include cutting and reshaping various waste products to make arts and crafts, cutting envelopes into smaller pieces to use them as scrap paper, etc.

Mechanical recycling

Mechanical recycling techniques can involve the use of grinding techniques to comminute the scrap material and produce recycled products in different size ranges suitable for reuse as fillers or partial reinforcement in new composite material (Pickering 2006). It can only be performed on single polymer plastic. Steps involved are:

- Crushing/Shredding/Milling
- Contaminant separation
    - Floatation
    - Washing and drying
    - Agglutination
    - Extrusion

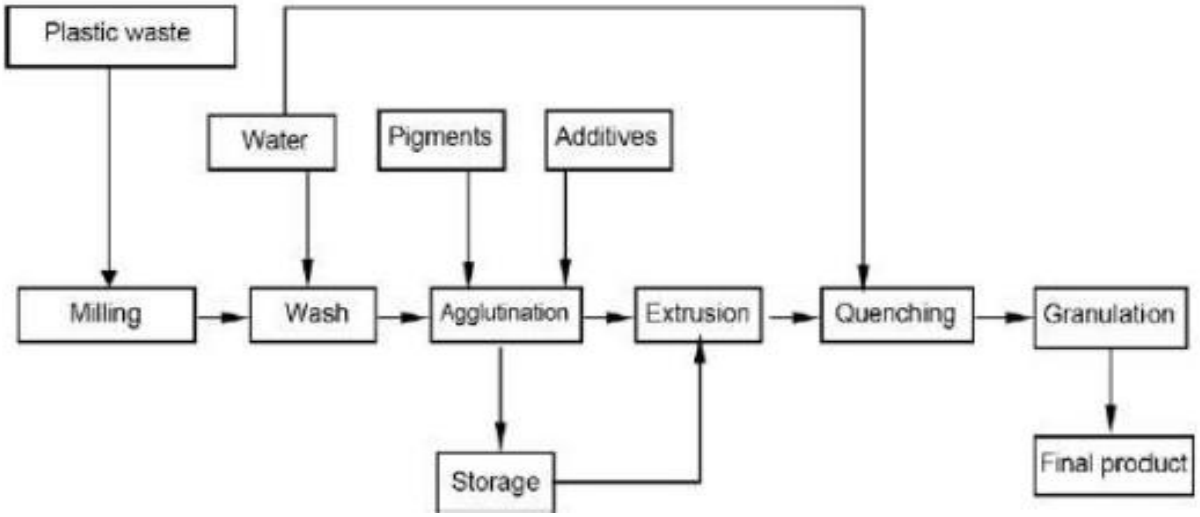


Figure 29 Mechanical recycling steps as described by Aznar et al. 2006

2.6.4. Tertiary recycling

Tertiary recycling refers to reprocessed by either a chemical process or heat in order to make the material/product reusable. Examples include melting metals, chemically treating old paper and breaking down plastic bottles in order to make brand new products (Recycling Consortium 2014).

Chemical recycling

An example is chemical recycling of PET, that has been more successful, as de-polymerization under milder conditions is possible. PET resin can be broken down by glycolysis, methanolysis or hydrolysis, for example to make unsaturated polyester resins It is converting waste into basic chemicals by pyrolysis, gasification or, as said, hydrolysis. It can also be converted back into PET, either after de-polymerization, or by simply re-feeding the PET flake into the polymerization reactor, this can also remove volatile contaminants as the reaction occurs under high temperature and vacuum (Hopewell, Dvorak, and Kosior 2009).

Thermal recycling

Thermal recycling processes involve the use of heat to break the scrap composite down and a range of processes are described in which there are various degrees of energy and material recovery (Pickering 2006).

2.6.5. Quaternary recycling

Energy recover

Quaternary recycling means recovery of energy from wastes. It is waste incineration to recover energy content as heat. It involves energy extraction by burning and incineration.

2.6.6. Other recycling processes

Multi-material recycling

The so called multi-material products, resultant specially from bigger requirements in products design, end up combining more than one material in the same product, which means a problem in its recycling process, due to the difficulty in separating their components.

The scrap material recycling consists of some basic actions that need to be followed or can be widely done separately:

- Collecting;
- Identifying;
- Separating;
- Sorting.

Frequently, the recycle of the polymers starts with a separation step categorized by density or thermal conductivity. Separation by density is the simplest way to identify the different polymers. Then the sorting of plastic wastes can be by various ways, such as:

- Density sorting;
- Hydrocyclones - using centrifugal force, enhance material wettability;
- Heavy medium separation - using tetrabromoethane;
- Triboelectric separation - sorting materials on the basis of a surface charge transfer phenomenon

Other ways can be pyrolysis, gasification or anaerobic digestion.

Micronization

Micronization appears as one of the technical applications to recycle materials without the need to separate by reducing the average diameter of a solid material's particles down to the micrometer or less. Traditional techniques for micronization focus on mechanical means, such as milling and grinding.

The results of the study made by Ashton et al (2016) showed that there was no degradation during the process, but recycled micronized LLDPE had lost ductility and tenacity on the one hand and increase in tensile strength on the other hand. In addition, intense decrease in the storage modulus in temperatures above 30 °C may limit the use of these materials in certain products. But generally the “micronization is a potential process to promote the recycling of multi-material products” and “resulting material has potential applications in new products” (Ashton et al. 2016).

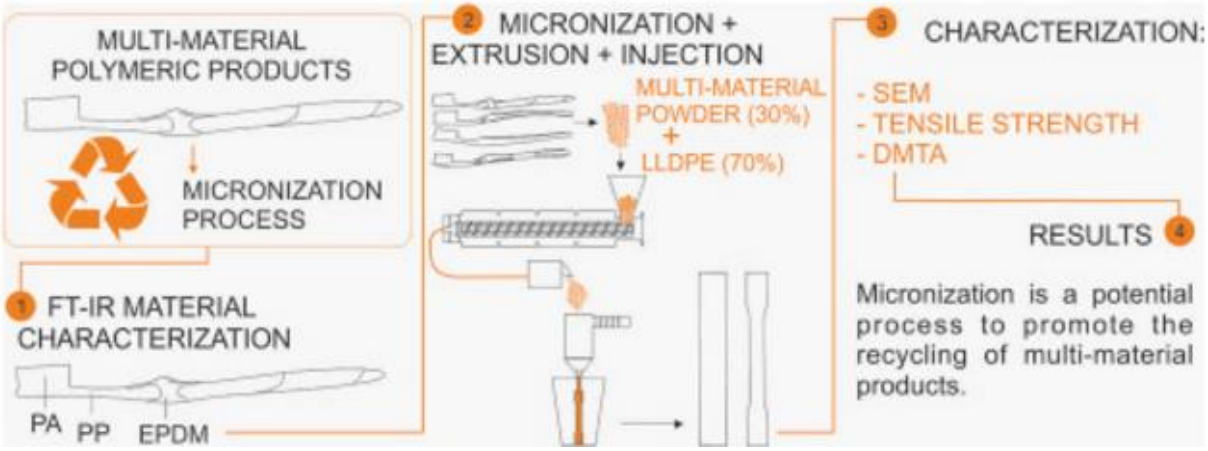


Figure 30 Micronization process (Ashton et al. 2016)

Aerobic composting

Citing other studies made on this field, i.e., (Cavalieri and Padella 2002) developed a composite material by a mechanic-chemical treatment of plastic waste, by a polymer milling process with liquid CO_2 that permitted to obtain a powder material that was successfully utilized as a matrix for a new composite material.

Controversy theories, such as the study from (Hopewell, Dvorak, and Kosior 2009) on which incineration with energy-recovery was indicated as the most suitable way for dealing with highly mixed plastic, go against the UK Resource Action Programme, shown on WRAP[12] report from (Michaud, Farrant, and Jan 2010). It indicates incineration as the second worst environmental performance, only after landfill, that despite that, is still the most common destination.

2.6.7. Biological degradation of plastic

Plastic degradation processes

- Photo degradation – degradation caused through the action of sunlight on the polymer;
- Composting – require specific levels of moisture and oxygen, and inadequate temperatures may not initiate the key hydrolysis reaction for PLA;
- Biodegradation – degradation that occurs through the action of microorganisms such as bacteria, yeast, fungi or algae;
- Biodeterioration.

The Japanese scientists from Technology Institute of Kyoto, led by biologist Shosuke Yoshida, recently announced (March 2016) the discovery of a microorganism capable of decompose the polyethylene terephthalate – PET – the plastic with which bottles are made, one of the most relentless solid pollutants on the planet.

The microorganism is one bacteria called *Ideonella sakaiensis* and was found on a garbage recycling plant feeding almost exclusively of PET, offering a more viable approach on the reducing efforts of the amounts of PET disposed on the environment. All that was known until then was some fungi capable of degrade PET partially, but using bacteria, as a way to develop a biological treatment for this type of waste should be much easier.

This works by producing two enzymes capable of degrading the molecular structure of carbon on PET's, which is particularly stable, on smaller components that can be incorporated on the environment without bad consequences.

The surprise on this finding was, according to scientists, that this bacteria appears to have acquired the ability to degrade this type of plastic in a process that lasted a few decades, which is pretty fast on the scale of biological evolution.

For now, a colony of these microorganisms can degrade a thin layer of PET within 6 weeks, which is particularly good compared to the centuries to degrade naturally. Now scientists are working on ways to industrialize the bacteria production so it could actuate on plastic debris that pollute the earth, especially on the oceans, (Yoshida et al. 2016).

[12] The Waste and Resources Action Programme: Material Change for Better Environment

3. MATERIALS USED

In this chapter it is described what were the materials used on this project, the main characteristics of them, and who were the supplier companies.

The materials used were recycled PVC and *Eucalyptus globulus* fibres, under the form of non-bleached cellulose pulp.

3.1. Recycled PVC

Plastics, also called synthetic resins, and are broadly classified into two categories: thermosetting resins and thermoplastic resins. Some examples of applications using these kinds of plastics are shown on Table 10.

Table 10 Plastic types and their applications, (Michaud, Farrant, and Jan 2010)

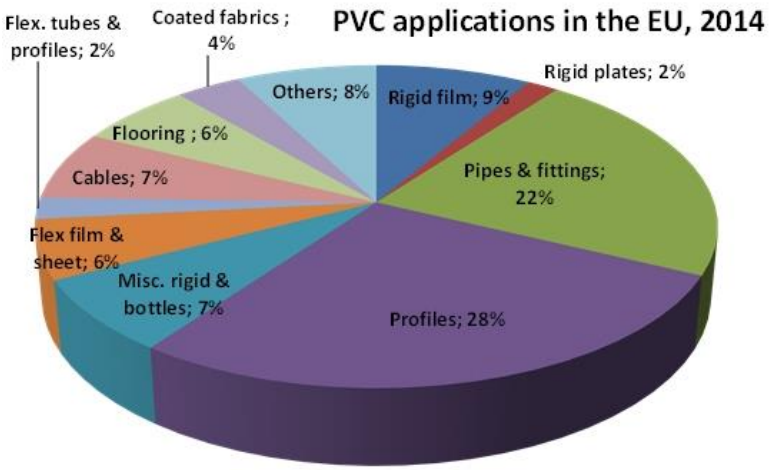
	Type	Use
Thermoplastics	PET	bottles, carpets and food packaging
	HDPE	bottles for detergents, food products, pipes and toys
	LDPE	cling-film, bin liners and flexible containers
	PP	yoghurt and margarine pots, auto motive parts, fibres, milk crates
	PVC	window frames, flooring, pipes, wallpaper, bottles, medical products
Thermoset	PU	coatings, finishes, mattresses and vehicule seating
	Epoxy	adhesives, sports equipement, electrical and automotive components
	Phenolic	ovens, toaster, automotive parts and circuit boards

Polyvinyl chloride, most known abbreviated as PVC, is among one of the three most produced and used synthetic plastics (Mulder and Knot 2001). A thermoplastic resin that can be recycled, since it can be re-softened by heating, contrary to the thermosetting resins that are not recyclable because their properties are degrade when they are heated up beyond a certain limit.

It is the most versatile plastic but mostly used in building construction industry. It appears into rigid form or into flexible form if plasticizers are added. The rigid form is mostly on pipes and fittings, window frames, but also in toys, transportation, packaging and medical applications while the flexible form of PVC is mostly applied on electric wiring mixed with other plastics, as well as on hot water and gas pipes. Examples of PVC are shown on Figure 31. Graphic 6 shows principal applications. Actually, PVC products offer good dimensional stability at ambient temperatures, resistance to chemicals and oils, durability, and a non-flammable nature.

(a) (a) (b) (b)

Figure 31 Applications examples of PVC: (a) rigid PVC; (b) flexible PVC (PVC.org, 2016)



Graphic 6 PVC applications on EU on 2014, (The European Council of Vinyl Manufacturers 2014)

However, PVC is one of the most toxic plastic and harmful to the environment. The presence of chlorine on PVC as primarily led it to various criticisms concerning health and environmental hazards, becoming considered inherently unsustainable, due to chorine chemistry association. However, the presence of chlorine imparts a range of unique technical features in PVC that set it apart from many other polymers (shown on the report (Leadbitter 2002)). The report cited some assesses made to the sustainability of PVC based on features such as its durability in use and the difficulty to break down. The evaluation model presented is based on The Natural Step (TNS[13]) framework, constituting the assessment to be made under a scientific basis, providing the means and necessary steps that are still missing on delivering PVC as truly sustainable polymer by the PVC industry.

The current considerable public concern about the problem of plastic wastes, from which PVC has not escaped, has a result on the gradually increasing quantity of used PVC items entering the waste stream. The material recycling or energy recycling may be a suitable way to overcome this problem. (Sadat-Shojai and Bakhshandeh 2011) report review considers the various methods of the PVC recycling and specific problems about some proposed processes, separation techniques, and recycling of mixed PVC wastes.

3.1.1. Sucatas DR

Due to its hazards factors, such as the presence of chlorine, PVC is sometimes not even put under consideration when it is about to recycle although it needs to be recycled like all the other materials. Findings for new applications for this material and the material characterization will ultimately increase its recycling and selling.

A search about what places in Portugal deal with recycled PVC was made. After a few contacts, it was scheduled the first reunion with an enterprise, located in Braga, called Sucatas DR - David Abreu Roque Unip Lda - that practices management and recovery of recyclable solid waste. Their main source is electrical wiring that they receive as considered refuse product.

On that first meeting, the objective was mainly to know the installations and visit the fabric to understand how do they deal with PVC. It was seen the process, since the reception of the discarded material from which they recycle PVC, until the final material that is the granulated recycled PVC that the enterprise sells.

[13] The TNS framework is a robust and science-based set of tools that define sustainability in unambiguous and workable terms and helps organisations engage with the practicalities of sustainable development.

They receive about 100ton of electric wires per month. The electric wires used by the company can be seen on Figure 32.

Figure 32 - Discarded electric wires for recycling at Sucatas DR

Electric wires are mainly consisted by metal parts at the interior such as aluminium and copper (alloys or not), and plastic isolate covering the entire exterior, as seen on Figure 33.

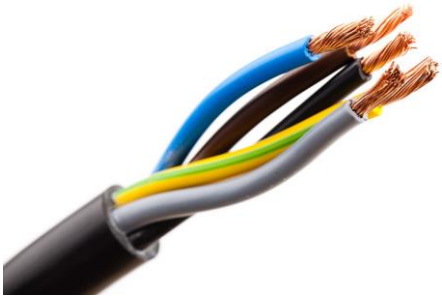


Figure 33 Electric wire - metal and plastic (Electric wire and cable, 2016)

With ambition, this company acquired the technology necessary to obtain the different components individually. The metal fraction is manually and easily separated from the plastic and are actually well put to good use, by melting and reusing. The plastic presented as an insulating cover largely considered a no value product is mainly constituted by polystyrene (PE), polyvinyl chloride (PVC) and a reticulated (with rubber). After separation from the metal, the plastic is all triturated. Afterward, the granulated plastic goes to a bath where because of low density, PE floats and is separated from PVC that is denser. PVC sinks and lies on the bottom of the tank, shown on Figure 34.

Figure 34 The metal tank where the plastics are separated by density

After separating the PVC from the PE, it is transported through air systems to big transportation bags, as seen on Figure 35.

(a) (b) (c)

Figure 35 (a) detail of air transport system from the tank; (b) air system transportation to the bags; (c) a full bag ready for transportation and another being filled

These bags go to the extruder support system, and the material enters on the extruder machine by gravity. The extruder has two big spindles, working at 200ºC, at such a speed that does not allow the PVC to grab the machinery and crown, as shown on. These extruded filaments are cut intro granules by rotating blades, what can be seen on Figure 36 and Figure 37.

(a) (a)

(b)

Figure 36 (a) Extruder feed and detail; (b) Extruder

(a) (b) (c)

Figure 37 (a) Detail of the extruder; (b) Cutting blades of the extruded material; (c) Extruder

When the filaments are cut into small granules they often get off the extruder stick one to another due to the high temperature that the material reaches on the extruder when passing through the spindles. To be detached they pass on the air system with a curve, where they shock and separate from each another. Figure 38 shows this system.

Figure 38 Packing system - air system transportation

The final material, granulated PVC, is mostly for extrusion and injection applications. Figure shows the result.

Figure 39 Granulated recycled PVC from Sucatas DR

All the wastes produced by the production sector are recovered and triturated, shown on Figure 40

Figure 40 Recovery/recycling system

3.2. Cellulose

To have a bio-composite one of the material must have biomass' origin. The Eucalyptus fibres were chosen once it is a natural and vey abundant source in Portugal, that causes a big waste stream contributing for fire propagation, and that could be used to reduce this risk and create value-added products.

Lignocellulose

Lignocellulose or lignocellulosic materials refer to plant biomass. It is the most abundantly available raw material on the planet being the most abundant organic substance. It is composed of three major constituents: cellulose, hemicellulose (carbohydrate polymers) and lignin (aromatic polymer), on percentages and structure represented on Figure 41. They combine to protect energy-storing sugars and give the plant cell wall strength and structure.

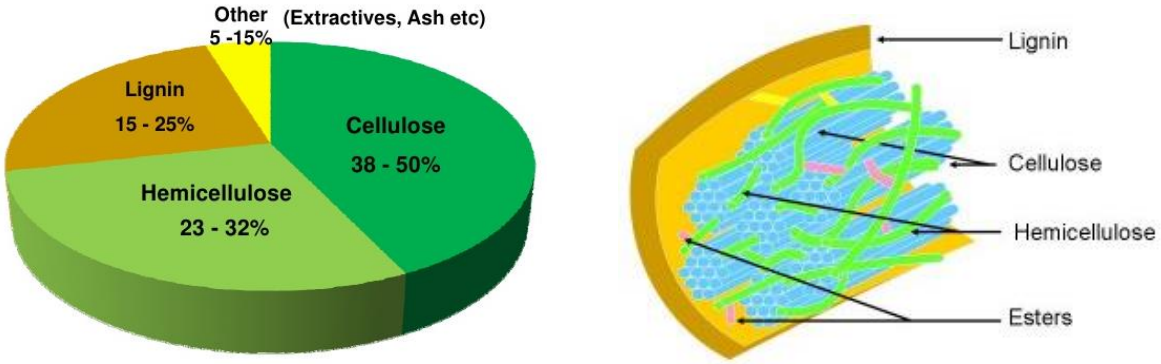


Figure 41 Representative scheme of a cell wall (Lignofuel Wordpress, 2010)

All plant species are built up of cells. If a cell is long in relation to its width, is called a fibre. A fibre has the shape of a microscopic tube of which the "void" is called the lumen. The cell wall, referred to as lignocellulosic fibre is made up mainly of cellulose, hemicellulose, and lignin.

Cellulose

Cellulose is a polysaccharide found in plants, the major component of natural fibres. A polysaccharide is a very large carbohydrate, consisting of many monosaccharides connected by o-glycosidic bonds.

The organisms in nature use polysaccharides for one of two purposes:

1) As a form of storing energy: animals store glucose on glycogen, and plants store energy on starch; both glycogen and starch are polysaccharides with alpha bonds forming a helical structure, perfect to work as energy storages;

2) As a structural and protection form: plants have individual glucose monomers linked together via beta glycoside bonds, forming a linear structure. This many and very long linear structure can stack on top of one another via hydrogen bonds forming a microfibril. This gives cellulose a very strong nature optimal for providing structure, protection and support to plants.

Therefore, cellulose is formed by hydrolysis[14] of beta glucose monomers, aligned or oriented by linear polymer chains consisting of thousands of linked anhydroglucose units. In cellulose, every β glucose monomer is upside down with respect to its neighbours. It means that in order to form a straight chain, each monomer must rotate 180º, (Richard and Sun 2011). On Figure 42 is represented the plant the cell wall, from which the fibres zoom out, reaching the glucose monomer.

[14] Hydrolysis is a reaction involving the breaking of a bond in a molecule using water

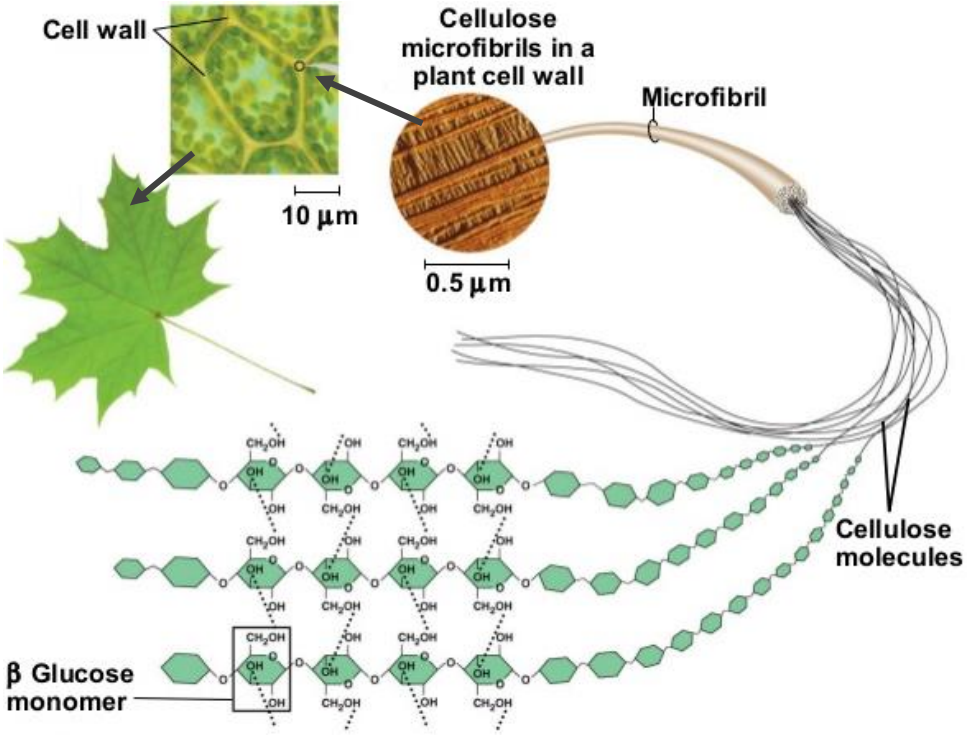


Figure 42 From a glucose monomer to a plant cell wall (adapted from The Molecules of Life, 2011)

Cellulose can be modified into cellulose esters, such as cellulose acetate, cellulose acetate propionate, and butyrate, which are currently used as major component thermoplastics.

Lignin

Lignin is an organic substance present on wood that binds the cells, fibres and vessels and that lignifies elements of plants. It is the most abundant renewable carbon source on Earth and the second most abundant natural polymer in the world, surpassed only by cellulose.

There are two mainly types of lignin: those which are sulphur-free, and the sulphur bearing ones that also include lignosulphonates and Kraft lignin which have to date been commercialized. Between 40 and 50 million tons per year are produced worldwide as a mostly non commercialized waste product. Due to the lack of suitable industrial processes, the sulphur-free lignin still is non-commercialized, (ILI 1992).

Chemically, lignin is a mixture of ramified natural polymers as a dendritic network of phenyl propene basic units, yet this substance appears with variations in its chemical composition and a great structural complexity, where any defined repeated unit does not exit, as shown on Figure 43.

Presented as a natural, renewable, obtainable at a reasonable cost, this raw material has got a strong substitution potential extended to products currently sourced from petrochemical substances, such as (ILI 1992):

- Multy-polarity related products / materials – as referred, lignin is a natural branched and crosslinked network polymer, which lends itself to use in materials, and contains both hydrophilic and hydrophobic groups. Specific treatments can strengthen either characteristic for particular applications as in emulsions and dispersants. On the particular case of this project the presence of lignin on the filler enforces the hydrophilic links (between PVC and cellulose) being the crude cellulose pulp with lignin preferable to the blenched pulp;

- High purity / value applications – lignin can be used as support materials for food and cosmetic applications including gels or emulsifiers or can work as an active substance with anti-oxidant, anti-bacterial and anti-viral properties;
- Agriculture – lignin and lignin derived products play an important role in the formation of soils and in plant and animal nutrition.

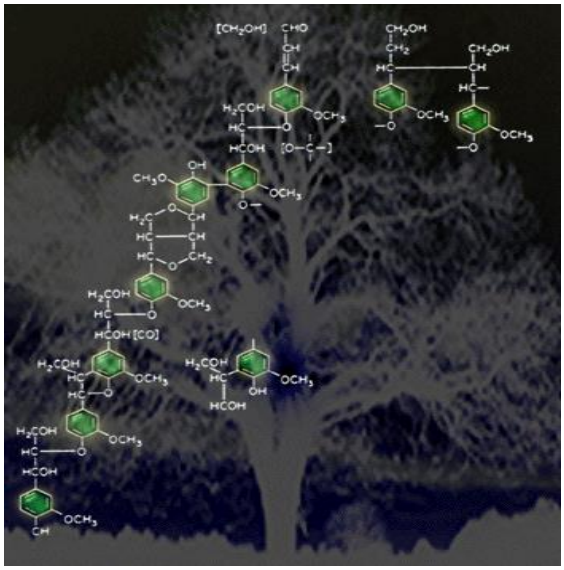


Figure 43 Examples for typical structures in lignin's chemical formula (ILI 1992)

Birgitha Nyström, research leader for materials technology at Swerea SICOMP said that there's no reason to believe that today's fossil-based carbon fibres can't be replaced directly with lignin carbon fibre in the large-scale production of lightweight materials, and the manufacturing methods for composites must therefore become more cost-effective.

Nanocellulose

Nanocellulose also referred to as microfibrillated cellulose, MFC, or nanofibrillated cellulose, NFC, is microfibrils released from the cell wall of cellulose, derived from wood fibres. Delaminating cellulosic fibres in high-pressure homogenisers produce it. A scheme is shown on Figure 44. Fully delaminated nanocellulose consists of long microfibrils with 1-2 micrometres length and 5-20 nm diameters and has the appearance of a highly viscous, shear-thinning transparent gel, as shown on (3dprint, 2015) Figure 45. It has exceptional strength characteristics on a par with Kevlar, a lightweight material used to manufacture high-strength, durable materials. However, in contrast to Kevlar® and other materials based on fossil fuels, nanocellulose is completely renewable, (Larsson et al. 2012).

Applications for nanocellulose

There are a wide variety of potential applications for nanocellulose, in the field of nanocomposites, non-caloric food thickeners, emulsion/dispersion, oil recovery applications, cosmetic/pharmaceutical applications. There are also applications in the electronics sector, strengthening agent in paper with a high filler content and other areas of application may be surface sizing and coating, e.g. as a barrier material (against oxygen, water vapour, grease/oil) in food packaging. Figure 46 shows a scheme showing the method process of obtaining NFC and NCC as well as some applications.

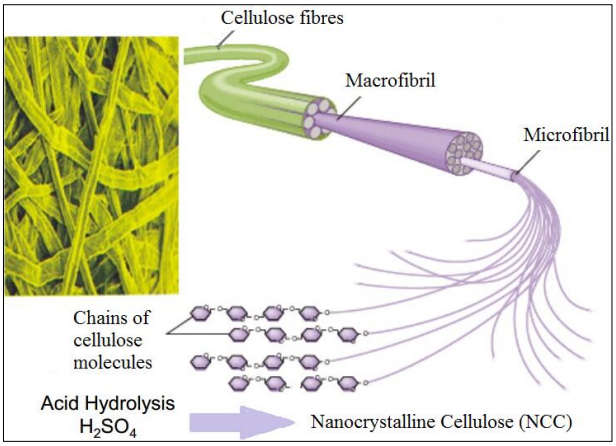


Figure 44 From cellulose fibres to nanocellulose (3dprint, 2015)

Figure 45 MFC gel (Innventia AB, Sewden)

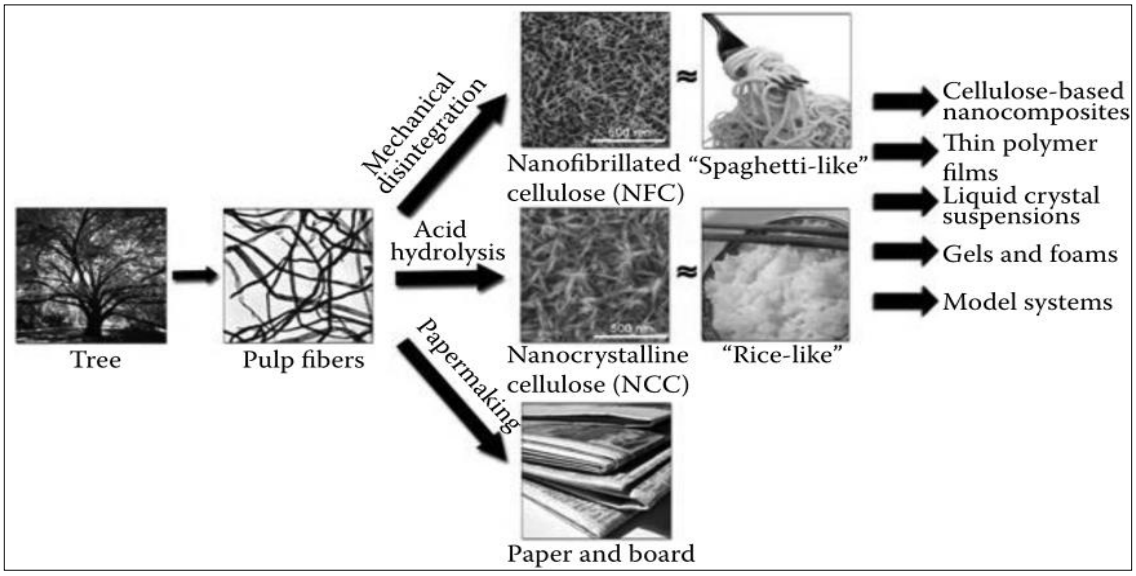


Figure 46 Potential uses of NFC and NCC (Notícias de Nanotecnologia 2014)

Theodora Retsina, the CEO of American Process Inc., says nanocellulose is as strong as carbon fibre and will rival it for 3D Printing, with lower weight, cost competitive, thermally stable at high temperatures, and blends with hydrophobic polymers.

3.2.1. The Navigator Company

The supplier of the fibres was The Navigator Company and RAIZ. The Navigator Company Group is a leading European manufacturer of uncoated woodfree printing and writing paper and one of the largest in the world of bleached eucalyptus Kraft pulp[15] (BEKP), generating the highest level of national value added, being the second leading exporter at Portugal, mainly to United States and Europa, within 118 countries over 5 continents. The Group manages 120 000 hectares of woodlands, mostly of *Eucalyptus globulus* (74%). The annual industrial capacity is 1.4. million tons of pulp of which 1.1 million tons is incorporated into paper and 2.5 TWh of electricity.

[15] Pulp is a lignocellulosic fibrous material obtained by separating cellulose fibres from wood.

The first contact with this company was established during a Workshop held at FEUP, about eco-efficient materials and sustainability. On that workshop, it was explained to the representative of the company the context of this project, on which he agreed to provide the fibres after a material transfer agreement was assigned by both parts - FEUP and RAIZ.

RAIZ, which is the The Navigator Company group's research institute for the forestry and paper industries, located in Quinta S.Francisco, Eixo, and works independently and networks with universities and other R&D units. Set up in January 1996, RAIZ is a private non-profit organisation, which helps the forestry and paper industries to be more competitive, through research, technological support and specialist training. RAIZ is a joint venture between the The Navigator Company group, the University of Coimbra, the University of Aveiro and the Higher Institute of Agronomy.

To better comprehend the manufacturing process of the Eucalypt fibre cellulose pulp, there was made a visit to RAIZ laboratories to understand the process, from the access of the eucalypt and their pests and plagues to the collection and process of the wood, until the final product, which is the cellulose pulp.

3.2.2. Manual obtainment of cellulose eucalyptus fibres

A technical fibre has its configuration depending on the fibre extraction method, which separates a bundle of elementary fibers from the stem or culm, as it is in the case of flax, bamboo, hemp, and others, (Bogoeva-Gaceva et al. 2007). Other plants like abaca, jute, sisal, hemp and others, still have manual extraction processes of the fibres, as shown on Figure 47, people still do it in a rustic way.

(a) (b) (c) (d)

Figure 47 Manually extracted fibres of (a) abaca; (b) jute; (c) sisal; (d) hemp

For this project there were also made attempts on manually extracting fibres from the eucalypts. The extraction of the fibres needed to be directly from the poles of *Eucalyptus globulus*. For this effect, they were collected on one of the places where the Portuguese companies go to harvest the *Eucalyptus* wood for the cellulose industry. This was at a private terrain with the owner consent, at Arouca county, Espiunca parish, the location is shown on Figure 48, and the place itself is shown on Figure 49.

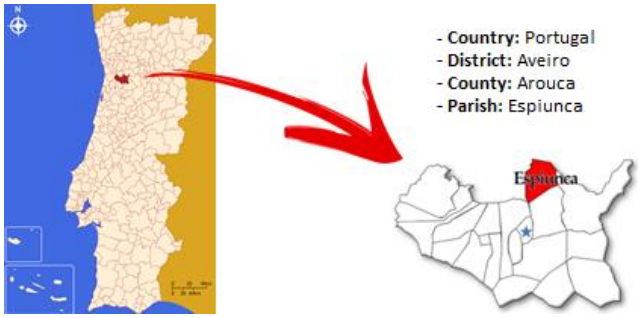


Figure 48 Location of Espiunca at Portugal

(a) (b) (c)

Figure 49 Espiunca: (a) Terrain perspective; (b) Detail of Eucalyptus poles; (c) Cutting of the eucalypt poles

As it can be seen, the field is full of branches, leaves and forest refuse, that create a huge forest waste and conditions for fire propagation.

As said before, the Eucalypt tree is a hardwood, so it is hard to work with and the manual extraction of the fibres would be such a challenge. Even so, first steps to achieve this extraction were made. First, the bark of the eucalyptus was removed once it has many of phenolic components that inhibit the adhesion to polymers. Simple pulling the bark had resulted in some cases, on others a snap-off blade utility knife was used to pull out the bark. Then, an attempt to extract the filaments from the poles as made, by cutting the poles into small pieces of wood for the interior to be more exposed and easily accessible, as shown on Figure 50.

(a) (b) (c) (d) (e)

Figure 50 Peeling of the eucalypt bark and cutting the poles for better fibre extraction

For the extraction of filaments, the tool shown on Figure 51 was made. The upper part that has got the pull and cutting function was screw to a wood support, that was fixed to a soft wood handle part. The tool's tooth have not a great resistance. After a few attempts of pulling out some filaments from the poles of the eucalypt hardwood, some tooth had broken up, making the tool unworkable for the purpose, as seen on Figure 51 (b).

(a) (b)

Figure 51 A tool to fibre extraction

Also, attempts on extracting fibres from dry and wet poles were made, once the extraction of other natural fibres are made crudely with its own natural wettability.

The filaments were getting out of extraction too weak and too much small in length to be possible to do some reinforcement with them, as seen on Figure 52.

(a) (b) (c)

Figure 52 Manual extraction of the fibres: (a) Dry pole; (b) Wet pole; (c) Filaments extracted

So, another tool was made, this time bigger and with stronger tooth they should be softer to better handling, permitting the filaments to have better shape and thickness. Although the results had better length, it was also a worthless effort once the filaments were too much irregular, as seen on Figure 53.

(a) (b)

Figure 53 Stronger toll to manual fibre extraction (a) and some results of the extraction (b)

After this experience it was concluded that the better for this project would be using the fibres obtain from the pulp mill, that despite the fact that they are very small, they are generally regular on size and could work better as a good good for a composite material.

Finally, only the *Eucalyptus* fibre cellulose pulp from RAIZ was used.

4. METHODOLOGY

For a shorter and faster writing and reading of the continuation of the practical part of this project, there are shortly present on Table 11 the abbreviations that will be used and the correspondence to the codification.

Table 11 Abbreviations names do the materials used on the project

ABBREVIATIONS	RPVC	RPCV_25CEL	LCEL	WCEL	P#	S#
	Recycled PVC	75% of RPVC and 25% of LCEL	Cellulose pulp with 2% of lignin	White cellulose pulp with no lignin	Number of the plate	Number of the specimen

For the mechanical characterization of RPVC, and then for assessing the influence of eucalyptus fibres on the properties of RPVC, the composite RPVC-Eucalypt fibres was assembled in different percentages, and then the samples necessary to the various tests were cut from assemblies on plates form. A first plan prepared out to make comparisons between different percentages, as seen on Table 12.

Table 12 Composite material composition

P#	LCEL	%	RPVC	%	T (°C)
1	0	0	4 kg	100	250
2	1 kg	25	3 kg	75	250
3	2 kg	50	2 kg	50	250
4	3 kg	75	1 kg	25	250

The choice of first choosing LCEL instead of WCEL relates to the fact that lignin was proven before to be a natural adherent between a polymeric matrix and *Eucalyptus globulus* fibres (Graupner et al. 2014). “In natural fibre composites, the chemical bonding at the interface usually occurs between the hydroxyl groups of cellulose and lignin on the fibre surface and functional groups in the matrix (e.g., maleic anhydride groups in maleic anhydride grafted polypropylene),” (Bogoeva-Gaceva et al. 2007).

After the evaluations of this materials conclusions should be taken to follow with other kinds of natural fibres and different percentages.

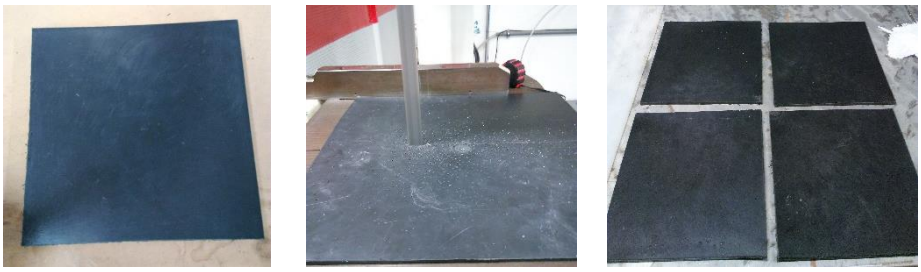
4.1. Specimens preparation

4.1.1. Recycled PVC and recycled PVC filled with *Eucalyptus globulus* fibres

Once the graulated PVC is prepared to be processed into a composite material, there should be machinery available to work with it. PVC is a kind of material that needs appropriate mechanical work for proper extrusion or injection. The working temperatures should be well controlled and machinery must have exaustion system because PVC can release cloridric acid when exposed to high temperatures, strongly dangerous to people and very corrosive.

At the metallurgical engineering department laboratories at FEUP there is an injection machine, that once was utilized to reprocess PVC. This experiment led to temperatures that reached the limits, what promoted the formation of chloric acid, damaging the machine. For that reason, PVC is not allowed to be used on that machinery. After this, there was made an agreement with the company that recycles PVC: they would make the plates with RPVC and LCEL at the company factory, where extrusion and injection machines are adequate to work with this kind of material, in exchange of the results of the tests that there would be realized, once they do not have any technical file of this material, it would be a great start for them to know some mechanical, acoustic and thermal properties.

So, the first plate, P#1, made by Sucatas DR, was a RPVC plate with 520 x 520 x 7 [mm], that was cut at FEUP into four plates with a saw for an easier manufacturing of the testing samples, shown on Figure 54.



(a) (b) (c)

Figure 54 Recycled PVC

The next plates, P#2 (plate number 2) and P#3 (plate number tree), were made of RPVC and *Eucalyptus* fibres on percentages of 25% and 50%, respectively. The preparation of the those are described on the following.

RPVC_25LCEL:

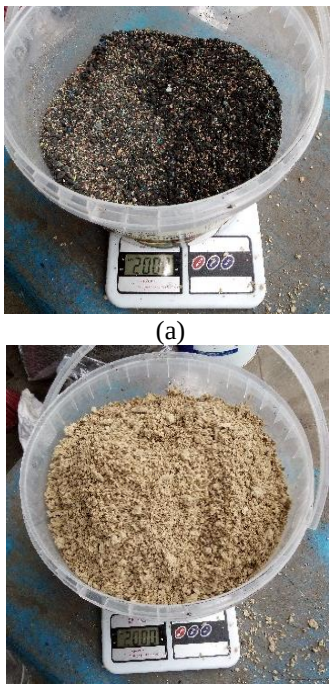
After putting the balance at zero by taking off the weight of the bucket container, it was weighted 3kg of RPVC and 1kg of cellulose pulp. The mixture went to the extruder screws where it got more homogenised, and then on the injection process at the press, the plate was finalized. The quantity of material was the enough to make the plate shown on Figure 55.

(a) (a) (a) (b)

Figure 55 (a) Measurements of the materials to make P#2; (b) Final P#2 composite: RPVC_25LCEL

50% LCEL:

Then the measures of the second plate were taken: 2kg of RPVC and 2kg of LCEL. The final result was the one shown on Figure 56.



(a)

(a)

(b)

Figure 56 Measurements for the P#3: (a) RPVC and LCEL; (b) Result P#3

The attempt on making a plate with 50% of LCEL (2kg) and 50% of RPVC (2kg) did not succeed. The adhesion between the two materials was poor, what led to a lack of capacity to fulfil the entire mould. Figure 56 (b) shows the result: on the centre of the mould there is some cohesion, but adhesion failures conduce to lack of material.

The mould, as seen on the P#1 made only with RPVC, is squared. One possible reason for the unsuccessful P#3 could be the lack of material, because of all the waste material that is lost along the machinery during the process.

Therefore, it was made a second attempt, where 3kg of RPVC and 3kg of LCEL were mixed. During the processes it was seen that a big amount of water vapour was being liberated from the machine through the exhaustion system, because the cellulose pulp stills brings water from the pulping processes.

For not damaging the machinery, it was agreed with the owners of the fabric to stop with the processes of production, especially the P#4 with 75% of LCEL, once the high quantity of water present on the cellulose pulp could damage the screws, for example.

Therefore, the processing of the composite with RPVC matrix and different percentages of LCEL was stopped ate this step and tests were made.

4.1.2. Recycled Polyethylene

The recycling of electric wires consists on separating the various kinds of plastics that make part of it. Since it is formed by polyethylene, polyvinylchloride and reticulated, it was seen an opportunity on recycling the polyethylene after separating it from the other materials. Some amount of that polyethylene was brought from the fabric, in Braga, to FEUP, for experience. Once this material had been mixed with PVC and was separated by floatation on water method, the same separation method was made again manually at FEUP, to ensure that no PVC was going to enter on the machine and damage it, as shown on Figure 57.

(a) (b) (c)

Figure 57 Granulated polyethylene (a); separation of PE by floatation method (b); injection machine – MIEQ, FEUP (c)

At the laboratories of metallurgical engineering department in FEUP, the material was injected but it hadn't enough consistency to fulfil the entire mould, as seen on Figure 58.

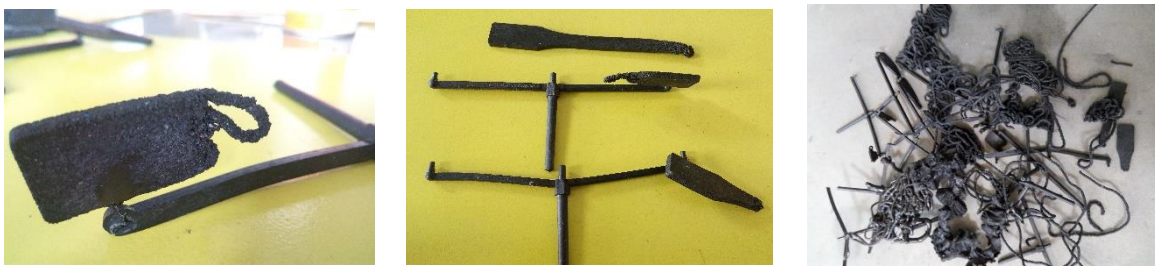


Figure 58 Unsuccessful PE tensile test specimens and waste material

There were defined various setting conditions such as injection speed, amount of material entering, and temperature (from 100ºC until 180ºC) but it was all ineffective and none of those variables lead to good results.

It happened because PE was contaminated by reticulated plastic materials, including rubber, detected by the smell of the injection process, what impeded of getting consistency on the final material, prompting to quite this experience.

Tests

At FEUP laboratories the specimens were cut from the plates and testes such as tensile and flexural testing, impact, shore hardness testing's, dilatometric, compression, sound absorption, reaction to fire, were made described on the following chapters.

5. RESULTS

5.1. Hardness tests

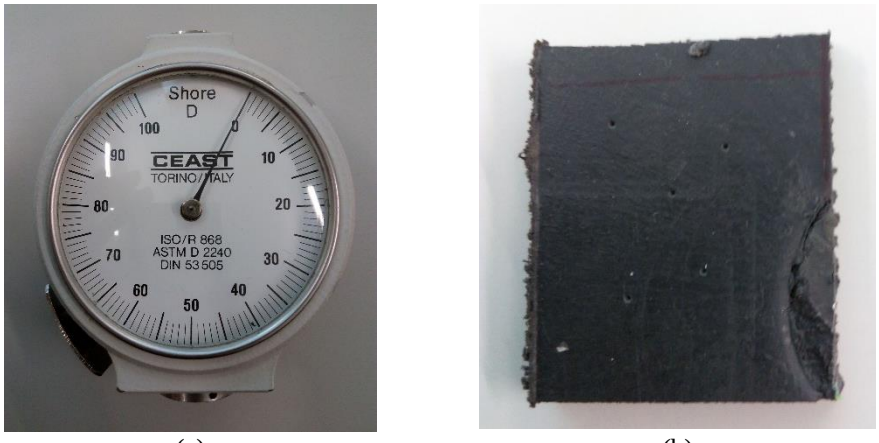
The Shore hardness measurements were made with an apparatus named as Durometer. The hardness value is determined by the penetration of the Durometer indenter foot into the sample, being the results obtained from this test a useful measure of relative resistance to indentation for various grades of polymers. Durometer and indentation can be seen on Figure 59.

Shore A and Shore D scale, as they are the preferred methods for rubbers/elastomers and softer plastics such as the vinyl. The Shore A scale is used for softer rubbers, whilst Shore D is used for harder ones.

Shore hardness test with Shore D scale were used.

Table 13 Hardness measurements values

	Hardness [Shore D]	
#Measure	RPVC	RPVC_25LCEL
1	45	46
2	45	45
3	45	44
4	45	50
5	44	50
6	45	45
7	45	46
8	44	48
Average	45	47



(a) (b)

Figure 59 Durometer (a) and specimen after being subjected to shore D test (b)

The presence of cellulose increases a little the hardness of the RPVC.

5.2. Tensile tests

The specimens and tests for the determination of tensile properties were machined according to the norm ISO 527-4, on annex 8, type 1B specimen, machined as shown on Figure 60.

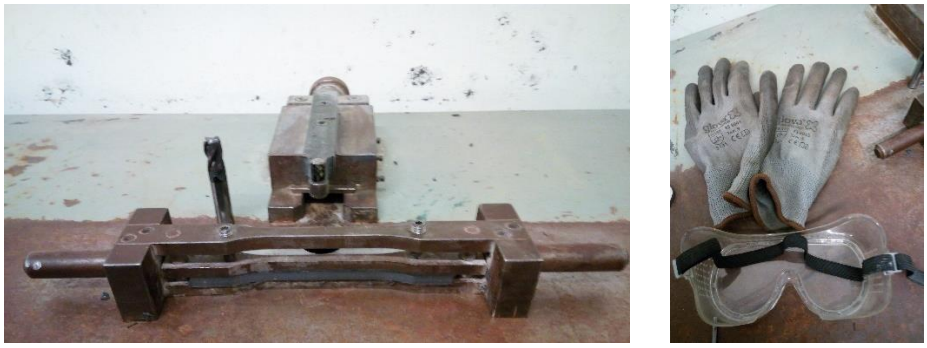


Figure 60 The machining of the specimens with the respective gloves and glasses of security.

Before the experiments, the width and thickness were measured in three points along the gauge, for the determination of an accurate value of the transversal area, where the force is applied.

The first RPVC specimen (S#1) was tested with an extensometer, as shown on Figure 61 (b), which was attached to the gauge length of the sample. The extensometer grips the sample and measures how much the sample is being stretched as the tensile forces on the sample are increased. Therefore, it allows to measure very accurately the extension on the sample as the load is increased. But due to the high ductility of RPVC it exceeded the limit of the extensometer, which only allows the extension until 50%. So the other specimens were tested without the extensometer so that the extension after the 50% of deformation could be registered.

The first RPVC_25LCEL specimen was also tested with an extensometer that lead to good results, once this composite material have less elongation than RPVC alone, so the experience continued using the extensometer in all specimens.

The tensile stress (σ_t), calculated with the maximum force registered during each experiment acting on the respective transversal area of the specimen gauge, could be calculated. The modulus of elasticity or Young modulus (E) was calculated considering the Hook's Law and according to the norm:

$$\sigma_t = \frac{F}{A} \tag{5.2.1}$$

$$\varepsilon = \frac{\Delta l}{l_0} \tag{5.2.2}$$

$$E = \frac{\Delta\sigma}{\Delta\varepsilon} = \frac{(\sigma_2 - \sigma_1)}{(\varepsilon_2 - \varepsilon_1)} \tag{5.2.3}$$

where σ2 and σ1 are the stress values for strain values given on the norm: ε1=0,0005 and ε2=0,0025.

The speed of testing for the first specimen was 2mm/min. The test lasted 15minutes. According to the norm the next speeds for specimen 1B is 10mm/min. So, speed test chosen to all the specimens was 10mm/min. The results are shown on Table 14 and Table 16.

(a) (b)

(a) (b)

(a) (b)

Figure 61 Tensile tests equipment and result specimens of (a) RPVC and (b) RPVC_25LCEL

Table 14 Measures of the specimens for tensile tests and tensile tests results for RPVC

#specimen	#measure	Width [mm]	Thickness [mm]	Transversal area [mm^2]	Maximum Axial Force [N]	σ_t [MPa]	E [MPa]
1	1	9,90	5,95	59,00	452,03	7,66	171,74
	2	9,95	5,95				
	3	9,90	5,95				
	Average	9,92	5,95				
2	1	9,95	6,0	59,90	458,35	7,65	90,78
	2	9,95	6,0				
	3	10,05	6,0				
	Average	9,98	6,0				
3	1	10,3	5,95	61,29	487,67	7,96	138,69
	2	10,3	5,95				
	3	10,3	5,95				
	Average	10,3	5,95				
4	1	9,85	6,05	59,82	407,23	6,81	80,48
	2	9,80	6,10				
	3	9,85	6,10				
	Average	9,83	6,08				
5	1	9,90	6,05	59,90	396,45	6,62	97,71
	2	9,85	6,05				
	3	9,95	6,05				
	Average	9,90	6,05				
					Average	7.34	115.88

The results on test made by Franco-Urquiza and Maspoch 2014, "*Viabilidad Del Reaprovechamiento de Residuos de PVC Provenientes de Cables Eléctricos: Propiedades Mecánicas*" say that the $\sigma_{máx}$ (MPa) calculated for that PVC varied according to the Table 15.

Contenido de residuo (%)	$\sigma_{máx}$ (MPa)
0	9.23 ± 1.03
7.5	5.91 ± 0.73
15	4.02 ± 0.35
25	4.07 ± 0.57
50	2.36 ± 0.35
75	2.43± 0.096
100	2.51± 0.33

Table 15 RPVC from Franco-Urquiza and Maspoch 2014

Table 16 Measures of the specimens for tensile tests and tensile tests results for RPVC_25LCEL

#specimen	#measure	Width [mm]	Thickness [mm]	Transversal area [mm²]	Maximum Axial Force [N]	σ_t [MPa]	E [MPa]
1	1	9,95	8,7	85,90	400,17	4,66	181,12
	2	9,95	8,55				
	3	9,95	8,65				
	Average	9,95	8,63				
2	1	9,95	8,7	85,88	507,38	5,91	216,2
	2	9,95	8,6				
	3	10,0	8,55				
	Average	9,97	8,62				
3	1	10	8,55	86,50	463,87	5,36	238,33
	2	10	8,65				
	3	10	8,75				
	Average	10	8,65				
4	1	9,8	8,8	84,79	487,32	5,75	237,58
	2	9,75	8,65				
	3	9,8	8,55				
	Average	9,78	8,67				
5	1	10,2	8,8	87,82	466,78	5,32	233,66
	2	10,05	8,65				
	3	10,15	8,55				
	Average	10,13	8,67				
6	1	9,75	8,7	86,13	432,51	5,02	167,33
	2	9,7	8,8				
	3	9,8	9,0				
	Average	9,75	8,83				
						5.34	212.37

The presence of 25% of LCEL on RPVC matrix improved the Young Modulus on 83%.

5.3. Flexural tests

The specimens for the determination of flexural properties were machined according to the norm ISO 14125, on annex 8.

Before the experiments, the width and thickness were measured in three points along the gauge, for the determination of an accurate value of the transversal area, where the force is applied. With those values, the stress could be calculated, as shown on Table 18. The speed of testing was 10mm/min.

The flexural stress (σ_f), calculated according to the following expression,

$$\sigma_f = \frac{3FL}{2bh^2} \tag{5.3.1}$$

Where

F is the load (N),

L is the span of the specimen (mm),

b is the width of the specimen (mm),

h is the thickness of the specimen (mm).

Table 17 Measures of the specimens for flexural tests and flexural tests results for RPVC

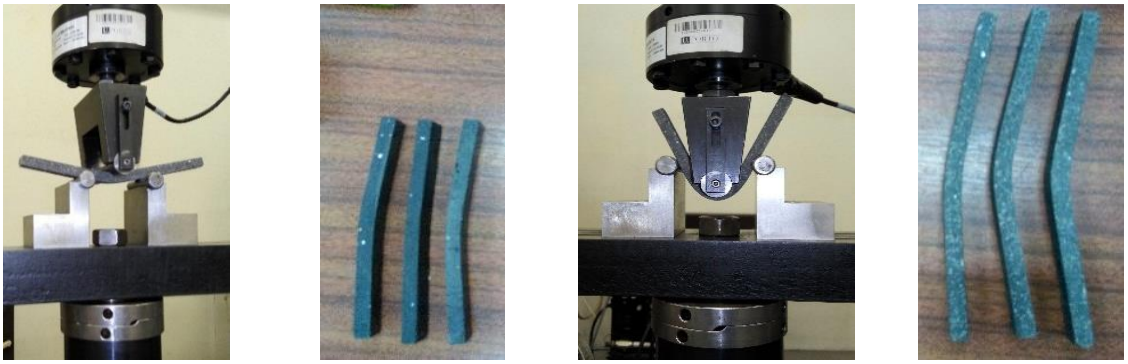
#specimen	#measure	Thickness [mm]	Width [mm]	Span [mm]	Maximum force [N]	σ_f [MPa]
1	1	7,3	14,3	80	55,2	8,5
	2	7,3	14,8			
	3	7,3	15			
	Average	7,3	14,7			
2	1	7,6	14,9	80	47,7	6,7
	2	7,6	14,9			
	3	7,6	14,9			
	Average	7,6	14,9			
3	1	7,4	15,3	80	54,9	7,8
	2	7,4	15,7			
	3	7,4	15,4			
	Average	7,4	15,46			
					52,6	7,7

Table 18 Measures of the specimens for flexural tests and flexural tests results for RPVC_25CEL

#specimen	#measure	Thickness [mm]	Width [mm]	Span [mm]	Maximum Force [N]	σ_f [MPa]
1	1	8,9	14,6	80	121,5	12,44
	2	8,9	14,7			
	3	9	14,8			
	Average	8,93	14,7			
2	1	9	14,7	80	133,9	13,31
	2	9	14,7			
	3	9,1	15			
	Average	9,03	14,8			
3	1	8,9	14,5	80	125,2	12,70
	2	9	14,6			
	3	9,1	14,7			
	Average	9	14,6			
					126,9	12,82

The presence of 25% of LCEL on RPVC matrix improved the flexural stress on 66%.

Figure 62 shows images of the flexural test while going on and the aspect of the specimens after the test.



(a) (a) (b) (b)

Figure 62 Flexural test equipment and result specimens of (a) RPVC (b) RPVC_25LCEL

5.4. Impact tests

The impact tests were made by means of a falling weight. To determine impact strength, three specimens of each material were machined. To fit the equipment, specimens were machined squared with 600 mm side. For each specimen three different impact energy values were chosen for the falling of weight, with a fixed mass of 3.742 kg. Figure 63 shows the instrumented falling weight impact tester (a) and detail of a specimen under testing (b). Table 19 shows the energy applied and the respective dropping height. Table 20 shows the results for the peak and the failure

(a) (b)

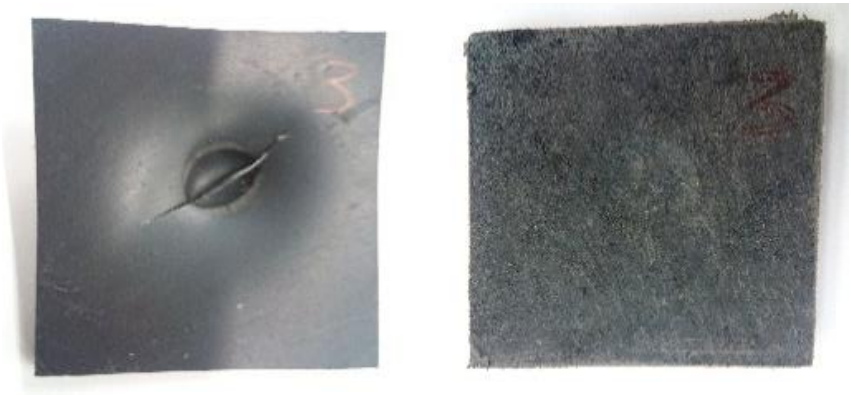
Figure 63 Instrumented falling weight impact tester (a) and detail of a specimen under testing (b)

Table 19 Energy applied to specimen's impact tests and respective high of drop

MATERIAL	#S	ENERGY	DROP HEIGHT
RPVC	1	10 J	0,270 m
	2	20 J	0,543 m
	3	40 J	1,088 m
RPVC_25LCEL	1	10 J	0,270 m
	2	20 J	0,543 m
	3	40 J	1,088 m

Table 20 Peak and failure values from impact test

		PEAK			FAILURE	
	#S	Deflection[mm]	Force[N]	Energy[J]	Deflection[mm]	Energy[J]
RPVC	1	7.666	1770.92	8.72	7.226	9.89
	2	10.004	2674.34	17.39	11.006	19.99
	3	10.668	3213.31	18.88	18.164	31.94
RPVC_25LCEL	1	3.895	2889.93	7.41	3.681	9.46
	2	6.186	3280.04	14.59	6.734	19.90
	3	7.113	4070.54	21.46	10.941	40.18



(a) (b)

Figure 64 Specimens #3 after the impact test: (a) RPVC with penetrating crack and (b) RPVC_25LCEL with very soft crack

5.5. Compression tests

The specimens and tests for the determination of compressive properties were machined, when possible, according to the norm ISO 604, on annex 8. According to this norm there are two types of specimens needed to characterize the compressive behaviour, one for modulus calculation and other for the stress calculation.

Figure 65 Photographs showing the cutting of the specimens for the compression testes

The specimens for the stress calculation were machined with 10*10mm with the thickness of the sample, while the specimens for the modulus calculation were machined with 30*10mm with the original thickness.

The machinery for the realization of the compression tests was the same used for the tensile tests but with different setting conditions.

The speed of testing was calculated accordingly to the norm, following the formula:

$$v = 0.02*l \ (=) \ v = 1mm/min \tag{5.5.1}$$

where l = 50 for modulus measurements

$$v = 0.5*l, \ (=) \ v = 5mm/min \tag{5.5.2}$$

where l = 10 for strength measurement with ductile materials, which yield.

The stress calculation is:

$$\sigma_c = \frac{F}{A} \tag{5.5.1}$$

Where

σ_c is the compressive stress value in question (MPA);

F is the measured force in question (N);

A is the initial mean cross-sectional area of the specimen (mm^2).

The compressive modulus calculation is:

$$E_c = \frac{(\sigma_2 - \sigma_1)}{(\varepsilon_2 - \varepsilon_1)}$$

where σ2 and σ1 are the stress values for strain values given on the norm: ε1=0,0005 and ε2=0,0025.

The results are shown on Table 21, where it can be seen that the presence of 25 % LCEL on RPVC matrix improves the compressive modulus.

Table 21 Results from the compression test

	#S	Stress σ_c (MPa)	Modulus E_c (MPa)
RPVC	1	198.51	22.51
	2	59.65	21.50
	3	53.61	18.82
RPVC_25LCEL	1	93.76	72.93
	2	206.59	62.68
	3	202.77	93.15

(a) (b)

Figure 66 Specimens after compression tests: (a) strength and (b) modulus

5.6. Dilatometry tests

Dilatometry tests of dilatometry consist on measuring the shrinkage or expansion of materials under a controlled regime of temperature and/or atmosphere.

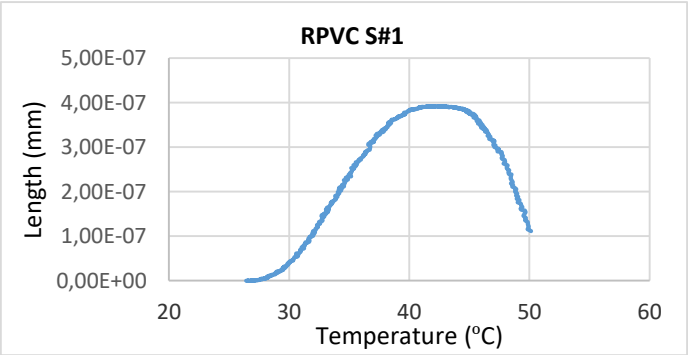
Specimens for this test should respond to the parameters on Table 22:

Table 22 Limits to specimens for dilatometry tests

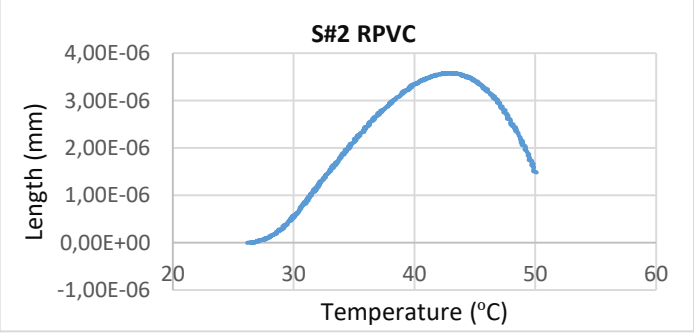
	Minimum	Maximum
Length [mm]	20	50
Width [mm]	5	10

Five specimens of each material with 30mm*8mm were machined but, because of the large affluence to the laboratory only two specimens of each material were tested.

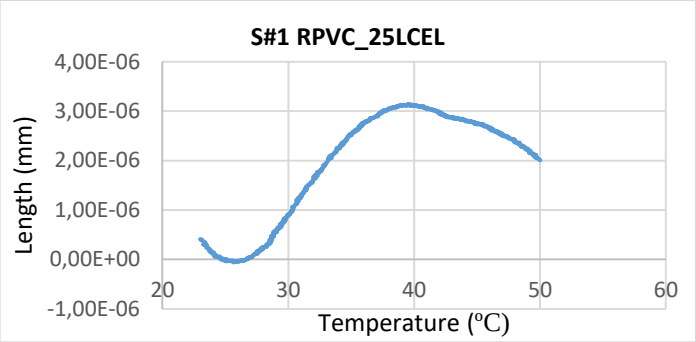
Since the decomposition temperature of PVC is about 180ºC, the maximum temperature chosen for this test was 100ºC, and the minimum temperature was the ambient temperature. It was a reasonable temperature to evaluate the behaviour of such material to heat, but far enough from damaging. However, the graphics originally given by the dilatometry laboratory, shown on annex 7 are not totally conclusive. It is supposed that the software used is not adequate to this kind of material after 50ºC, consequently the graphics were only evaluated until the temperature of 50ºC. On the following graphics, (from Graphic7 to 10), it can be seen that until 50ºC the results are pretty acceptable on specimen number 1 and number 2 of RPVC. Yet, if two specimens could be sufficient to analyse RPVC, (considering, even though, the little dimensions of the specimens and the non-uniform distribution of the different phases), the same is not applicable to RPVC_25LCEL material on which two specimens were not enough to cover a good average behaviour of the material.



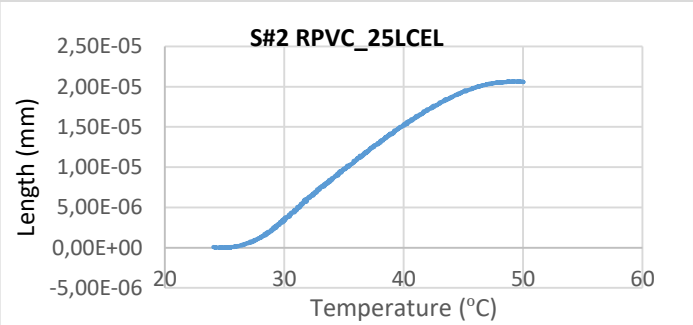
Graphic 7 Dilatometry curve of RPVC Specimen #1



Graphic 8 Dilatometry curve of RPVC Specimen #2



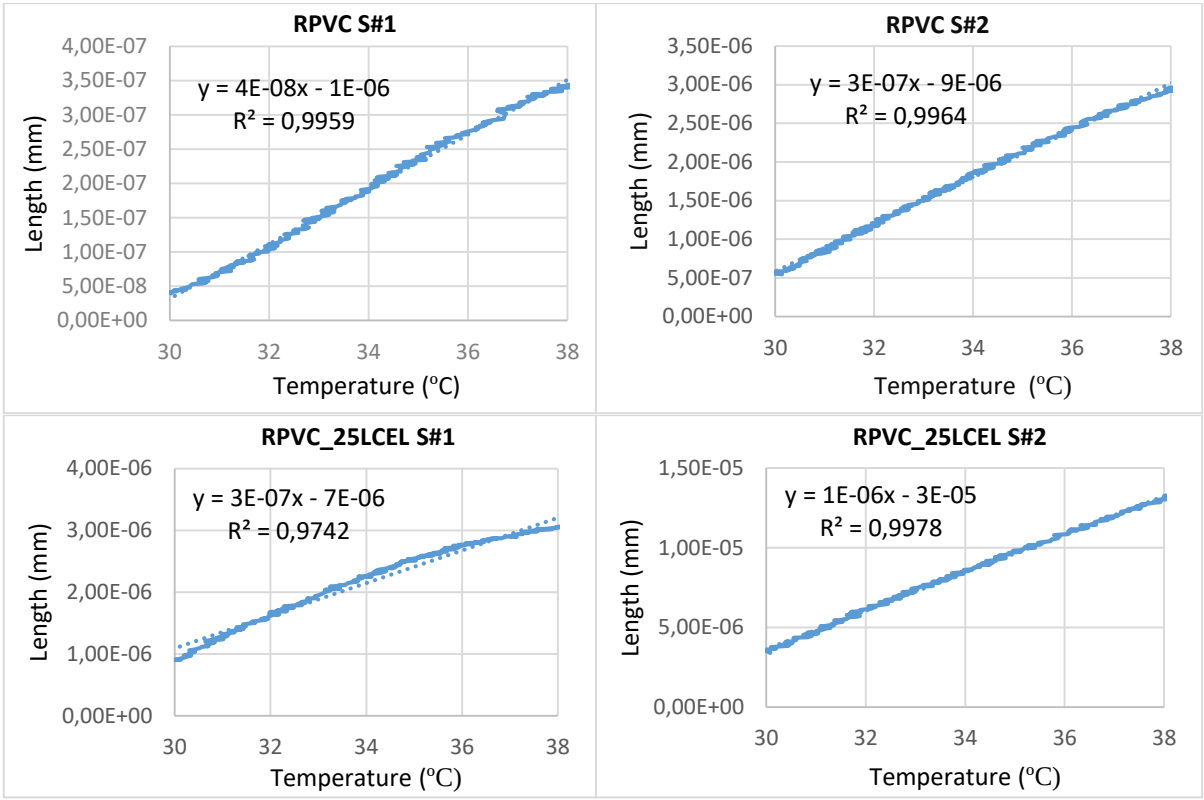
Graphic 9 Dilatometry curve of RPVC_25LCEL Specimen #1



Graphic 10 Dilatometry curve of RPVC_25LCEL Specimen #2

Although the mixture between the matrix and the fibres was homogenised to have a good composite, the RPVC matrix was still randomly filled with *Eucalyptus* fibres. On a very first and simple analysis, it could be assumed that specimen number 1 of RPVC_25LCEL can have more percentage of the matrix (RPVC) than the specimen number 2, which might have more percentage of cellulose than the first.

Considering that the points very close to the initial temperature are sometimes varied with the sample accommodation, it would be worth to consider the quantitative evaluation of data between 30 and 38°C, just before the the dilatometry softening is reached, around 40ºC. The dilatometry coefficient is given by the slope of the curve, shown on Graphic 11 and Table 23.



Graphic 11 Determination of the dilatometry coefficients

Table 23 Dilatometry coefficient

	Dilatometry coefficient $\overline{\alpha}_D$
RPVC	1.70E-07
RPVC_25LCEL	6.50E-07

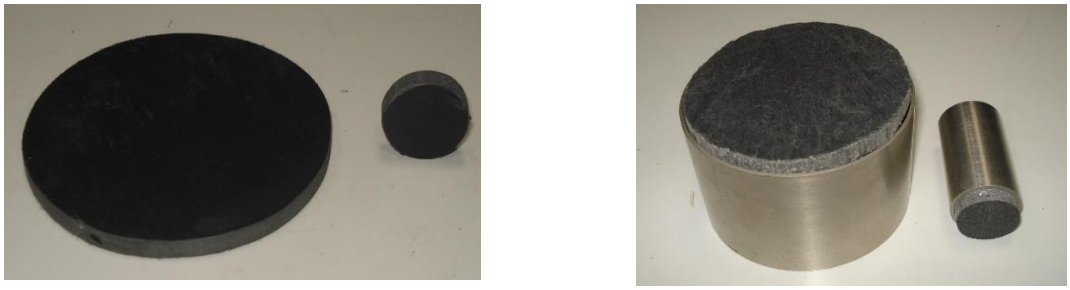
Also, there should be worthy to make this analysis with other material formulations, such as other percentage of cellulose on RPVC, and take more conclusive evaluations on the influence on *Eucalyptus* fibres on recycled PVC.

5.7. Acoustic tests

The equipment used and the procedure of the acoustic tests agreed, where applicable, with the specifications in the applicable standards of the norm ISO 10534-1: 1996.

This test consisted of the experimental determination of the sound absorption coefficient, taken by measuring maximum and minimum sound pressure levels, using the Standing Wave Tube method, with different frequencies for each sample being studied.

Specimens with diameter of 100mm and another one with 30mm were machined, as shown on Figure 67, for determination of sound absorption coefficients (α_s) at the octave band from 125 Hz to 2000 Hz, with the equipment Brüel & Kjaer model 4002 shown on Figure 68. The results are shown on Table 24. The graphics comparing the sound absorbance coefficients for different frequencies of this materials compared to concrete, which is the most common used building materials, are shown in annex 6.



(a) (b)

Figure 67 Specimens for sound and water absorbance tests: (a) RPVC; (b) RPVC_25LCEL set up on the support of the Standing Wave Tube

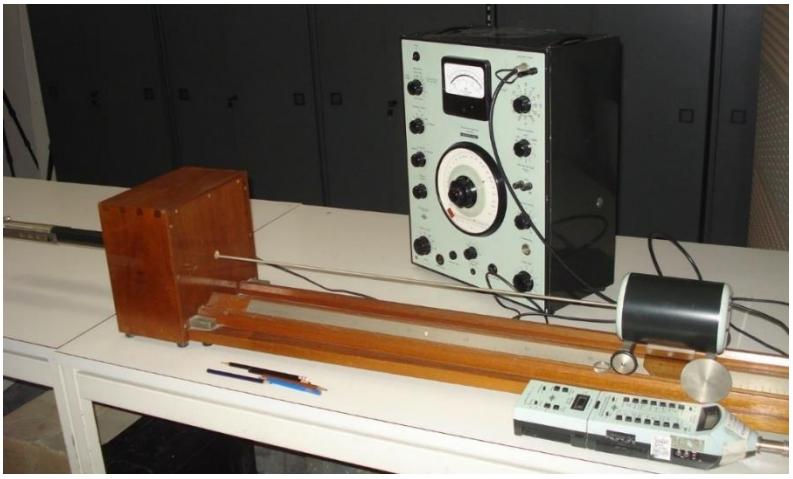


Figure 68 Sound absorption equipment: standing wave tube Bruel & Kjaer, model 4002

The equipment used for this experiment is the following:

- Signal Generator: brand Brüel & Kjaer, model 1024 (serial number 299575);
- Tube standing waves: brand Brüel & Kjaer, model 4002 (serial number 383904);
- Sound level meter precision: brand Brüel & Kjaer, model 2231 (serial number 934499 checked in ISQ - verification certificate - Cert No. 4578.);
- Set of 1/1 octave filters: brand Brüel & Kjaer, model 1613.

Table 24 Sound absorption coefficients

FREQUENCY (Hz)	SOUND ABSORPTION COEFFICIENTS (α_s)	
	RPVC	RPVC_25LCEL
125	0,06	0,06
250	0,11	0,14
500	0,15	0,16
1000	0,18	0,21
2000	0,14	0,16
4000	0,24	0,25

5.8. Permeability test

Permeability test that are usually made on tiles at civil engineering department was adapted to determine whether water can penetrate a RPVC and RPVC_25LCEL.

Once the determination of the acoustic coefficients is not a destructive test, the same specimens were used to the permeability test.

In this test, a dam is set up on the top surface of the specimens by gluing the bottom edge of the water source to the material, ensuring that the assembly is completely isolated to the dam. Then the enclosure is filled with water. After 2-hour period observation should be made to see if any drops were form on the underside of the specimens.

Figure 69 Set up of permeability tests on RPVC and RPVC_25LCEL

As expected, no drops were observed from which was concluded that the materials are very impermeable to water absorbance and water passing,

5.9. Reaction to fire

Reaction to fire test determines the contribution of a material to fire progress. The test consists on a single flame test where parameters such as the time of ignition or the destroyed length of the material are measured.

The specimens machined for this test followed the norm ISO 11925-2, with the size of 250*90[mm], and shown on Figure 70.

(a) (b)

Figure 70 Specimens for reaction to fire test of (a) RPVC and (b) RPVC_25LCEL

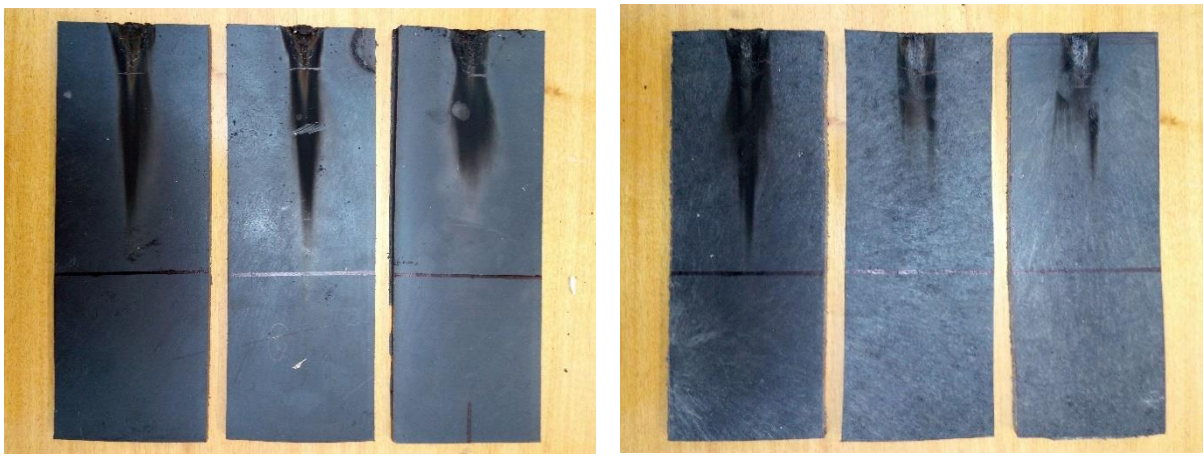
The specimens had been under conditioning for a week, at 23ºC and controlled humidity. The conditioning treatment followed the norm EN 13238. The classification follows the norm NP EN 13501-1. The single flame test equipment with the insider burner and the respective exhaustion, as well as the specimen installed on the support and the burner, are shown on Figure 71. The results, with the main parameters required by the norm and measured by an accurate observation method, are shown on Table 25. The aspects of the final specimens after the test are shown on Figure 72-

(a) (b)

Figure 71 Single flame test equipment (a) and ongoing single flame test (b)

Table 25 Results from the reaction to fire test

Material	#P	Exposition	Application of the burner (s)	Ignition (s)	Destroyed length (mm)	Extinction (s)	Falling drops	Time to reach 150mm
RPVC	1	Edge	30	4	28	32	No	Does not reach
	2	Edge	30	3	25	31	No	Does not reach
	3	Edge	30	4	30	31	No	Does not reach
RPVC_ 25LCEL	1	Edge	30	3	20	31	No	Does not reach
	2	Edge	30	5	22	31	No	Does not reach
	3	Edge	30	6	24	31	No	Does not reach



(a) (b)

Figure 72 Specimens after single flame test: (a) RPVC and (b) RPVC_25LCEL

5.10. Scanning Electron Microscopy

Samples of the materials used on this study were taken to the Scanning Electron Microscopy (SEM).

Due to the difficulty on getting access to this test, this opportunity was seized to analyse various samples of materials shown on Figure 73: *Eucalyptus globulus*, the eucalypt bark, the RPVC composite material with 25% of fibres content, the composite material with 50% of fibres content, and also a sample of G*enista Cytisus*, collected in the same place of the eucalypt.

The tree *genista* is a Portuguese shrub, known for being a very abundant of the Portuguese forest, and often considered a weed. Is it also known as the Portuguese broom, once their branches are used to make artisanal brooms. On the process of the forest cleaning to reduce forest fire risk, this tree should also be harvested once it constitutes a strong natural fire propagator, together with other biomass. This is often considered forest waste when harvested, because it has no commercial value. For these reasons, *genista* is a possible source of value-added products creation. Once this project has focused on Eucalypt fibres, the results shown on this chapter are the ones that referred to this fibre, while the results related to *genista* are shown in annex 5.

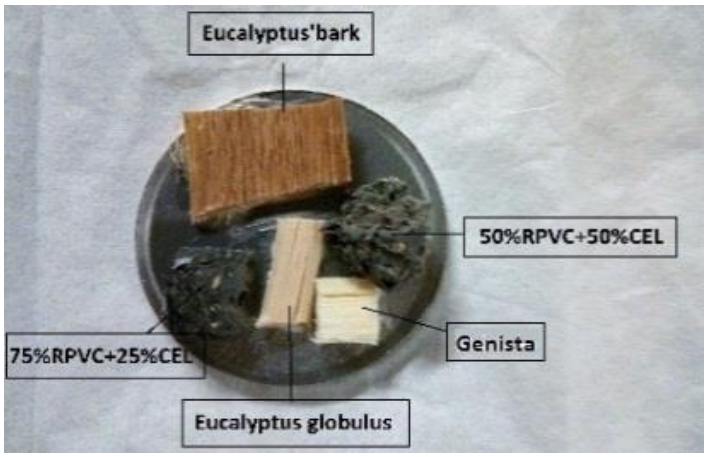
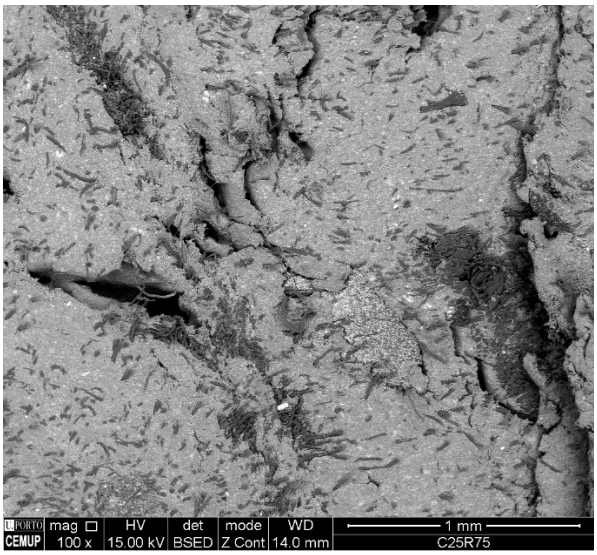
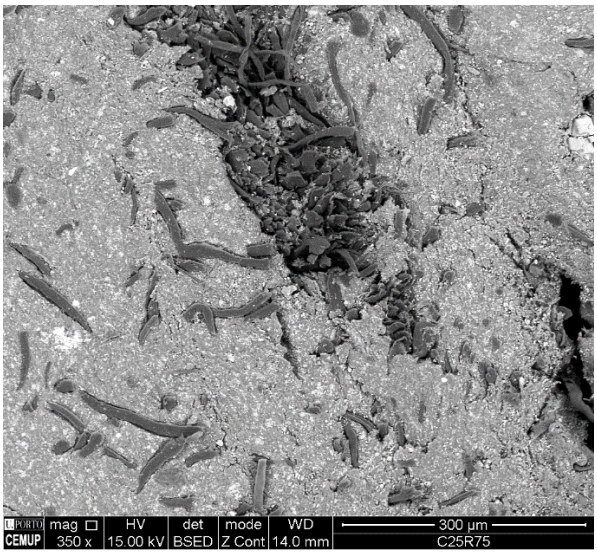


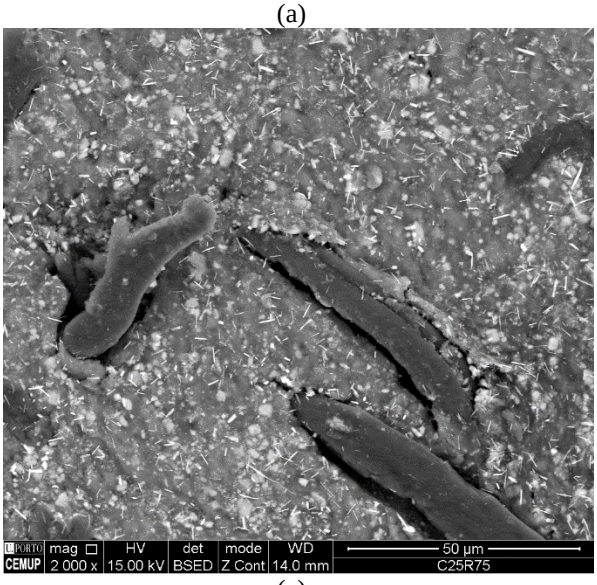
Figure 73 Samples for SEM analysis



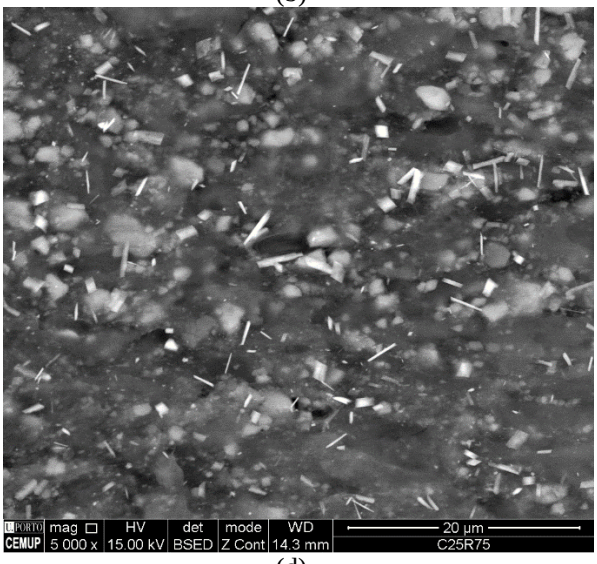
(a)



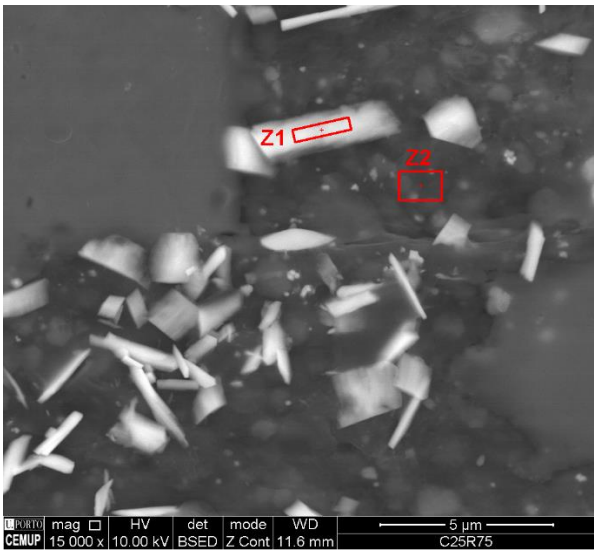
(b)



(c)



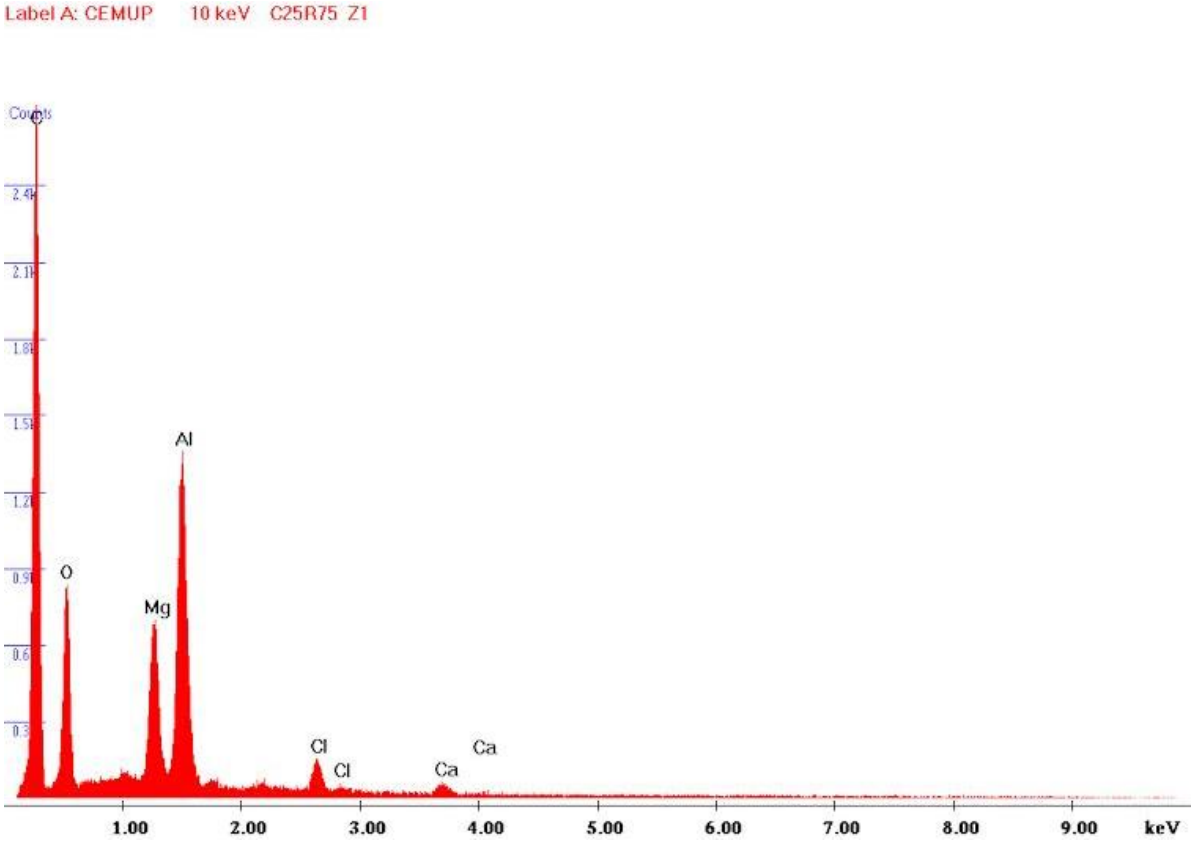
(d)



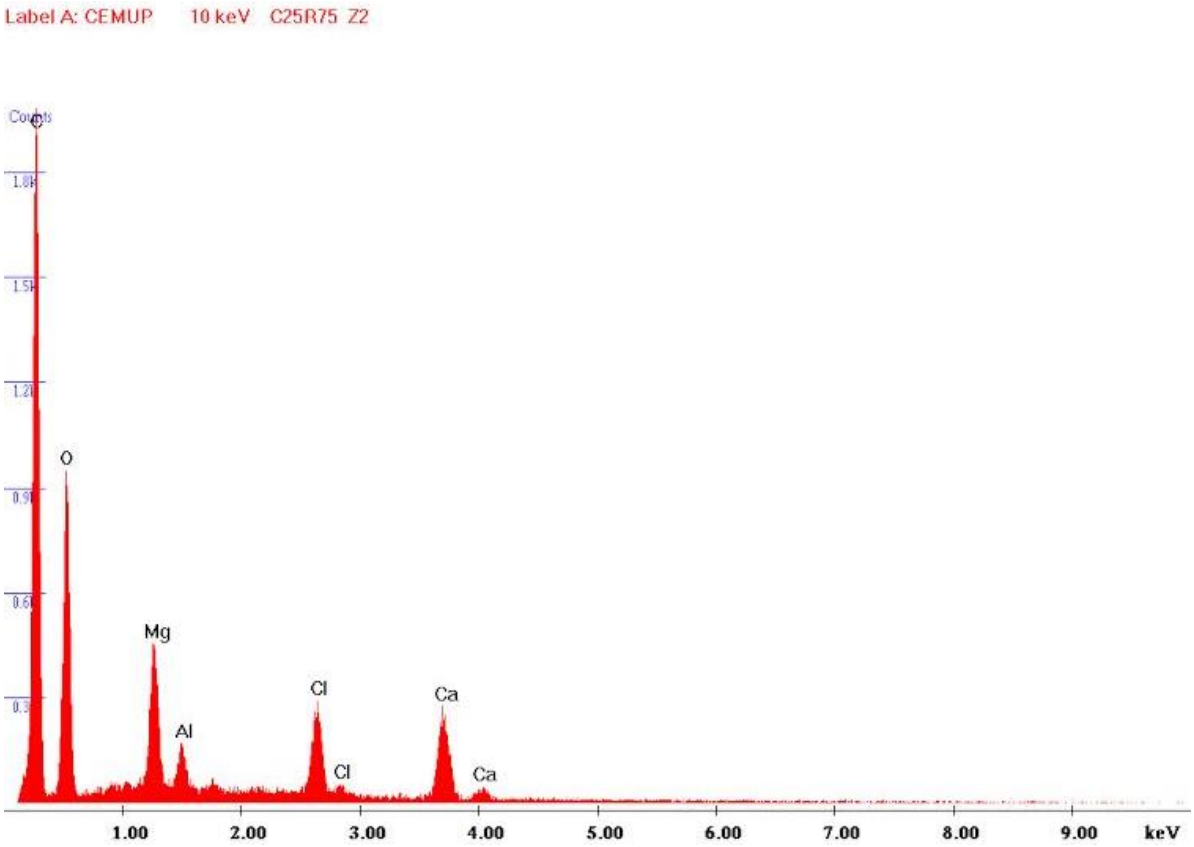
(e)

Figure 74 SEM images of the RPVC_25LCEL, scale: (a) 1mm; (b) 300 µm; (c) 50 µm; (d) 20 µm; (e) 5 µm

In Figure 74 (e), the most zoomed image, two different zones were marked, zone 1 (Z1) and zone 2 (Z2), which were subjected to energy dispersive X-ray analysis (EDX), that is an analytical technique used for the elemental analysis or chemical characterization of a sample, shown on Graphic 12 and Graphic 13.



Graphic 12 EDX spectrum from Z1 of RPVC_25LCEL



Graphic 13 EDX spectrum from Z2 of RPVC_25LCEL

The Z1 can be considered a metal residue present on the RPVC matrix, while the Z2 can refer to a matrix of PVC contemplating, or not, a fibre.
On the study of (Kumar 2015)**,** morphological analyses indicate poorer fibre adhesion to the matrix with increasing of the fibre content of epoxy and showed that there is poor fibre/matrix adhesion.

6. CONCLUSIONS

The context

While the legislations are becoming more tied about the industrial pollution and ecology issues such as circular economy and sustainable development, more industries are starting to have outlooks towards a greener tomorrow, by the substitution of fossil fuel derived components, pollutants and limited resources, for renewable sources or sustainable resources, and biodegradable components. There is an increasing replacement of metals and wood with composites, e.g., allowing structures to shed weight and consequently reducing the fuel consumption and the use of petrochemicals and CO_2 emissions. Among those composites there are emerging developments on bio-based materials, natural fibres and resins, along with cleaner and more efficient processes. The environmentally responsible researchers all over the world thankfully continue with many developments taking place in the green composites industry, in which the present project fits with the making of eco-bio-composite.

The project

Eucalyptus fibres have been used as a reinforcement material in polymer composites, due to its low density, abundance, low cost, and good properties. In this project, the composite material with recycled PVC and eucalypt pulp fibres was compared with its matrix solely. It has been observed that the composite material presents a greater hardness, higher impact strength, better dilatometry properties and acoustic isolation and better fire resistance; though the increasing of the fibre content lead to worst fibre/matrix adhesion, possibly due to the large amount of hydroxyl groups in cellulose that give hydrophilic properties to the natural fibres.

Cellulose is the main structural element of natural fibres and it is strongly polar due to hydroxyl groups, acetal, and ether linkages (C–O–C). This renders cellulose more compatible with polar, acidic, or basic groups, compared with nonpolar polymers such as thermoplastic and thermosets. Natural fibres are hydrophilic and have low moisture resistance, whereas many matrices are hydrophobic, which leads to poor interfacial compatibility between the two phases

6.1. Future approaches

Material characterization

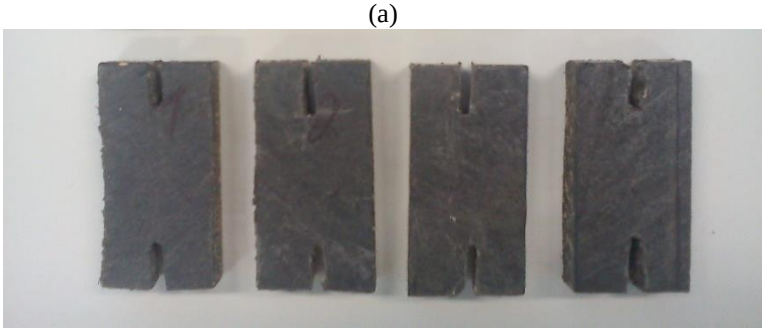
For a more complete understanding of the influence of the natural fibres of *Eucalyptus globulus* cellulose pulp in the mechanical, thermal and acoustic properties of waste PVC from electric cables, different percentages of this natural filler on the composite material, should be studied. This percentage of fibre content should not be less than 10%, according to (Kumar 2015) because with very little percentages of fibres, the load might not be homogeneously distributed over the material, and the matrix tends to absorb the maximum load, what can lead to the failure of the specimen by the matrix while the carrying of the load by the fibres is almost negligible.

It would also be interesting to make other comparisons with the same eucalypt cellulose pulp but with some pre simple treatments, like dry it in an oven before the assembly to the matrix, so that there is no shrinkage or voids after the water escapes. Other reason to do this treatment is to promote better adhesion between the matrix and the fibres, once the attempt of making a composite with 50% of RPVC and 50% of LCEL (per mass) was not well succeeded for the reasons said before (the higher quantity in fibre content the more difficult is the adhesion).

Abrasion tests

Specimens for this test were machined, as shown on to fit the equipment available. Unfortunately, the machine was occupied until a date after the finishing of this project, what led the tests to be done later.

(a)



(b)

Figure 75 Specimens for abrasion tests: (a) RPVC; (b) RPVC_25LCEL

Fibre-matrix adhesion

Relating to the content and treatments of the fibres there are some controversial theories. Ones say some superficial treatments of the fibres, with lignin for example, as shown on (Graupner et al. 2014) could improve adhesion of the fibres to the matrix for increased fibre content, while others say that, in general, fibres with higher cellulose content lead to better mechanical properties, because cellulose provides uniform molecular orientation that increases the adhesion to the matrix (Kumar 2015). Still, the main disadvantage of natural fibres in relation to composites are the poor compatibility between the fibre and the matrix. To enhance the fiber–matrix interfacial strength of natural fiber composites, it is necessary to use a physical or chemical treatment to change the surface structure of the fibers as well as the fiber surface energy, having effects on physical interactions and practical adhesion.

Material valorization

On a study made by (Pleissner et al 2013) on valorisation of hemp wastes in fermentative lactic acid production, hemp were taken into account as source of creating value-added chemicals. “Due to the high content of carbohydrates (≈58% dry weight), mainly consisting of cellulose and hemicellulose, hemp shives can be considered a potential and inexpensive source of fermentable sugars for the production of high value-added compounds, such as lactic acid”. Once *Eucalyptus Globulus* also have a significant percentage of cellulose and hemicellulose, studies should be carried out to use eucalypts as a carbon source in lactic acid fermentation after thermo-chemical and/or enzymatic hydrolysis.

Rubber

At the beginning of the organization of this project, we had in mind was on mind to work with the recycling of tyres, once they are one of the biggest sources of non-biodegradable waste that ought to be contoured, and more reasons referred on Chapter 1 INTRODUCTION. Some time was dedicated to search on this area through information gathered from BioSafe[16], meetings with students from the Master in Product and Industrial Design together with the Civil Department at FEUP and architecture professors, and also by meetings with Sucatas DR company (in which they have shown that they tried before matching recycled rubber with recycled PVC, but the material was of poor quality and all of those attempts were left behind because the material had no consistency to lead to good results concerning the field of construction). Though, BioSafe have been leading the environmental market in Portugal, showing that it possible to recover and apply this material, research and innovations are needed to keep following and turn over the tyre graveyards problem.

Figure 76 Tyre graveyards at Sulabiya (photo by: Mohammed Alsultan, 2012)

[16] BioSafe is a Portuguese company that provides crumb rubber solutions, by working in the production of new raw materials and products from End of Life Tyres (ELT), having a production capacity of over 25000 tons of ELT per year that is an equivalent of about 3.2 million of car tyres.

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8. ANNEXES

8.1. Annex 1 Comparison between linear and circular economy

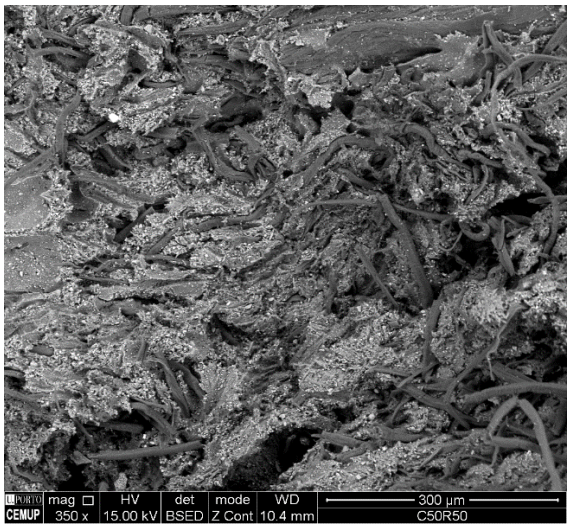
Comparison between linear and circular economy, (Hieminga 2015) (ING Economics Department)

	Characteristics of a linear economy		Characteristics of a circular economy
Attitude towards nature	Forcing nature to produce more	➔	Doing more with what nature can produce
Attitude towards production	Take, make and waste	➔	Reduce, reuse and recycle
Closing loops	One lifetime use of products, components, materials and energy	➔	Materials and energy flow infinitely in cycles through the economy
Product life extension	Products become obsolete while they are still usable	➔	Product life is extended in new applications or products serve as valuable inputs for other products
Performance economy	Consumers buy goods	➔	Accessibility and performance instead of ownership are leading in many consumption markets. Consumers increasingly share products
Earnings model	Producers determine sales price of products	➔	Producers charge price for the use of the product
Multiple values and principles	Money is the dominant value in business models	➔	Business models are based on multiple values (financial alongside environmental and social values)
Supply chain	Companies improve efficiencies in isolation of each other	➔	Companies work together to increase value along the supply chain. Risk and benefits are shared upstream and downstream

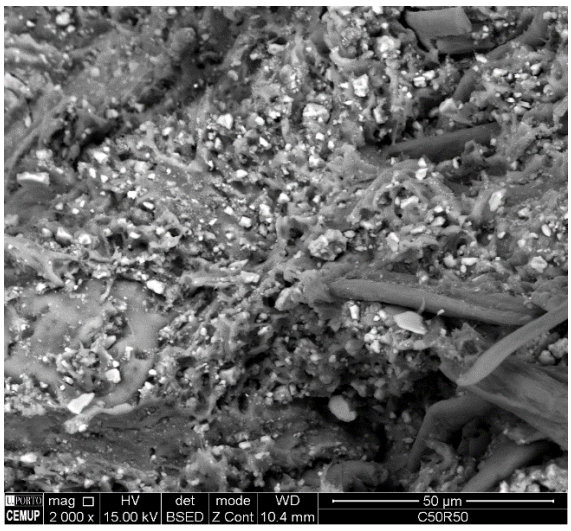
8.2. Annex 2 SEM images of RPVC_50LCEL

SEM images of RPVC_50LCEL scale: (a) 1mm; (b) 300 µm; (c) 50 µm; (d) 20 µm

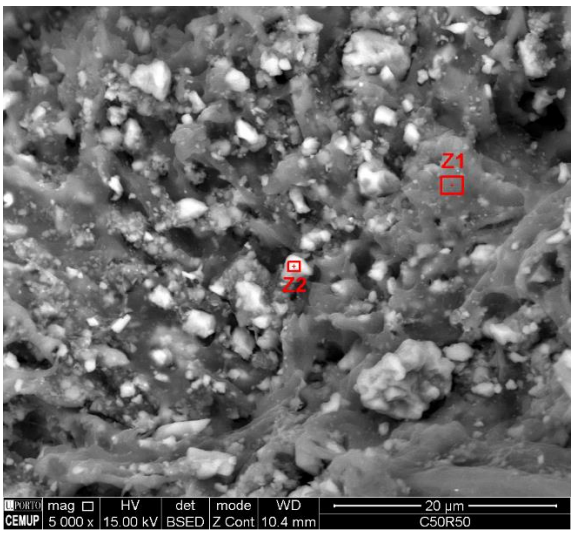
(a)



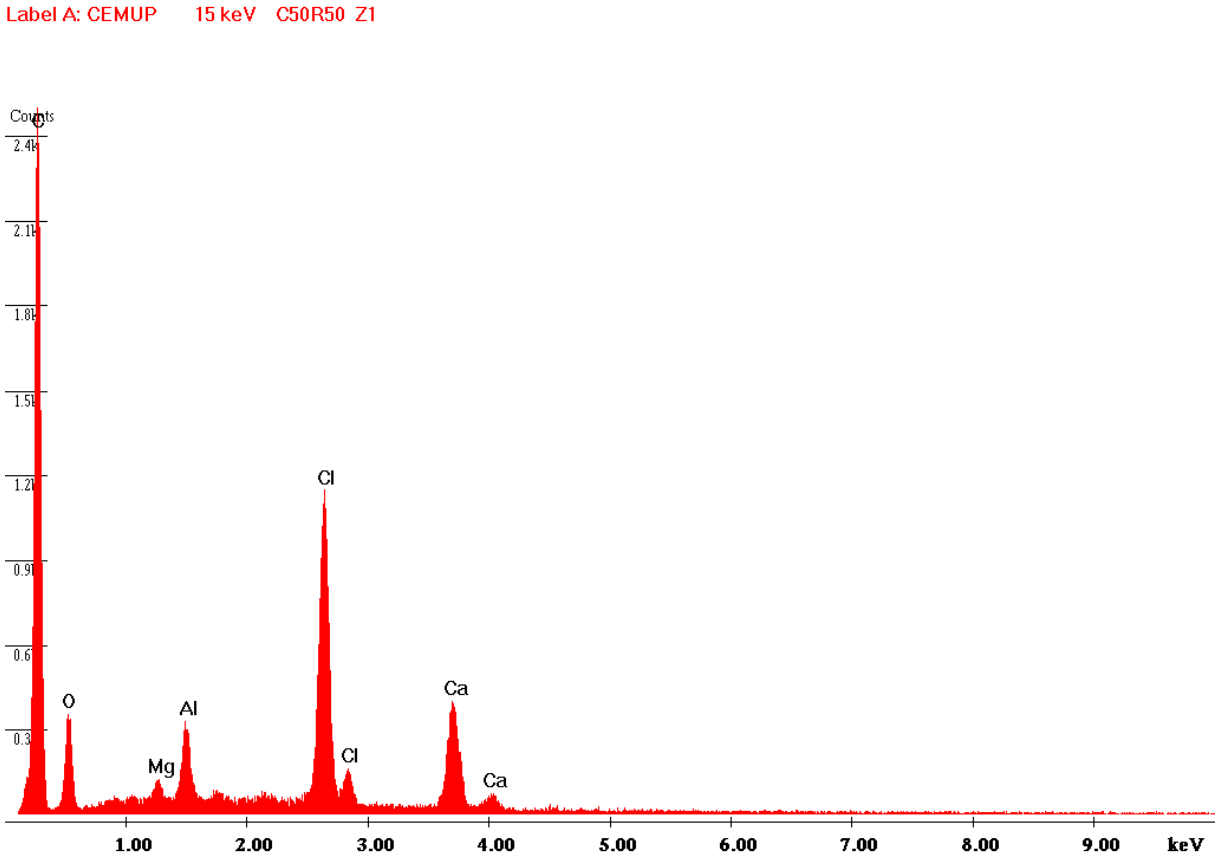
(b)



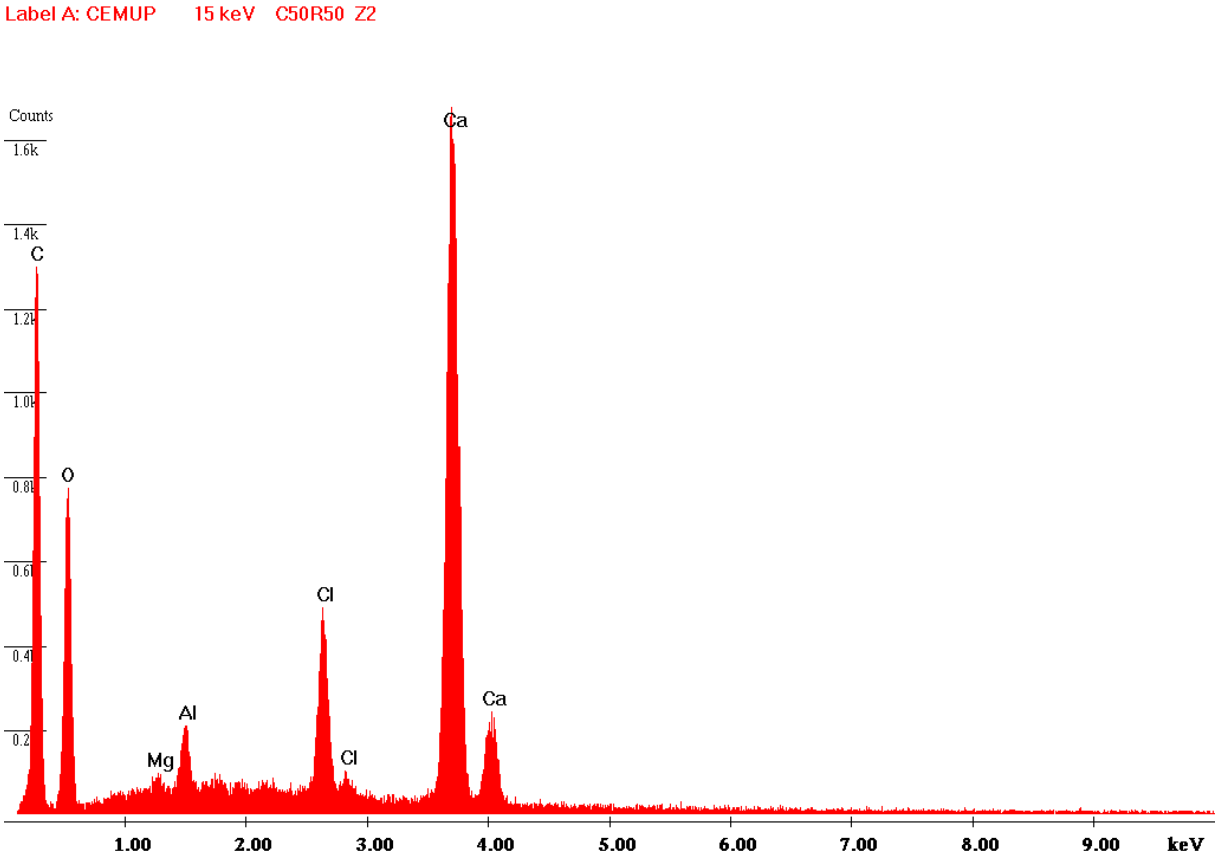
(c)



(d)



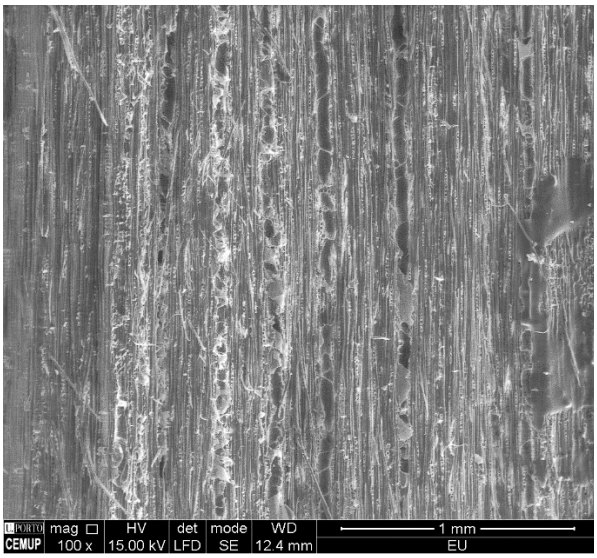
Constitution of Z1 from RPVC_50LCEL



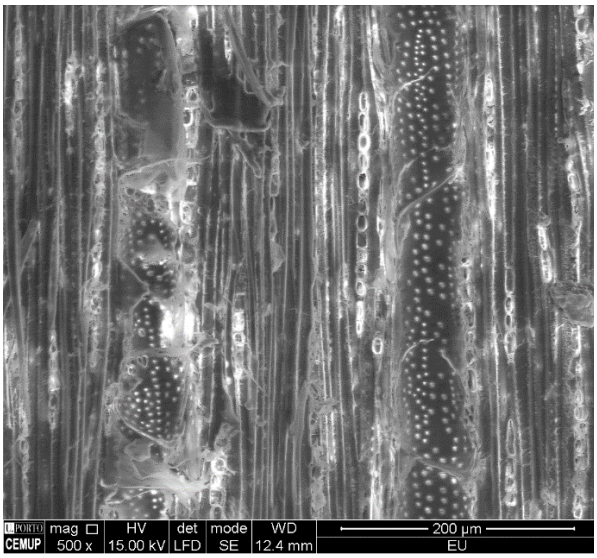
Constitution of Z2 from RPVC_50LCEL

8.3. Annex 3 SEM images of *Eucalyptus globulus*

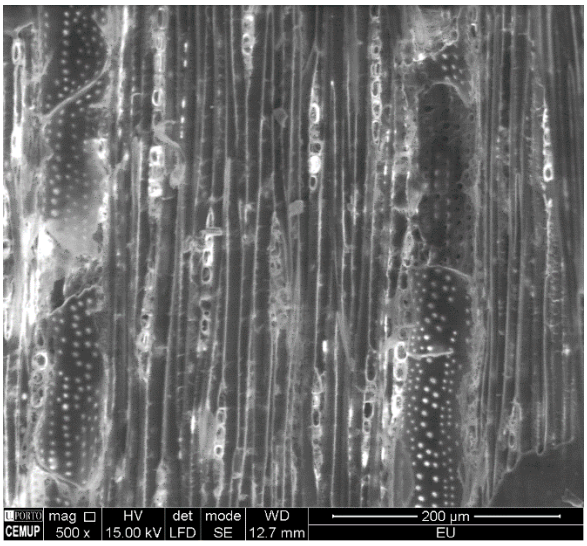
SEM images of *Eucalyptus globulus*. Scale: (a) 1mm; (b) 200 µm; (c) 200 µm; (d) 50 µm



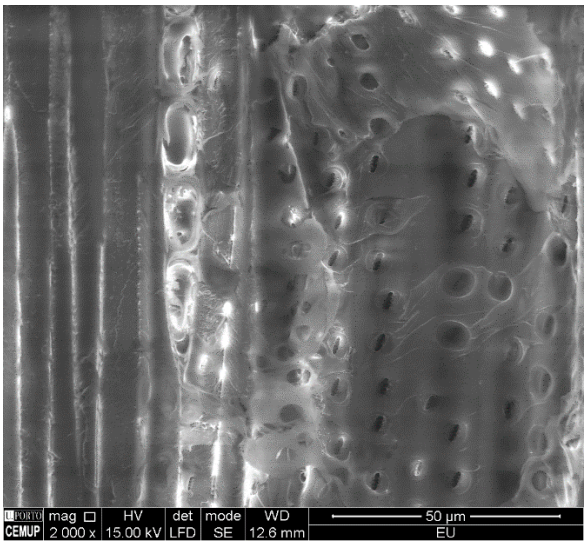
(a)



(b)



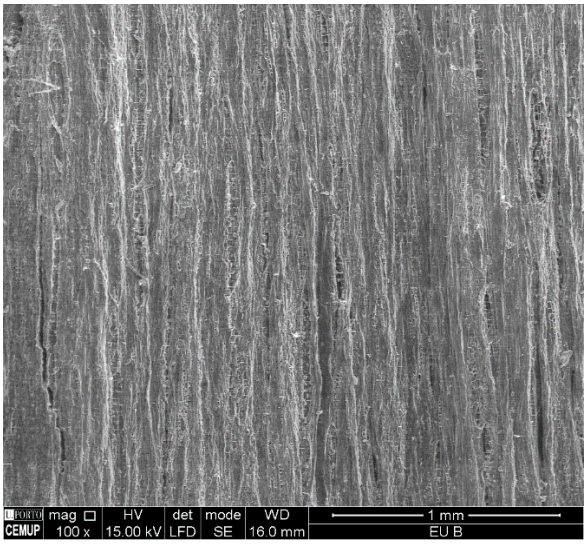
(c)

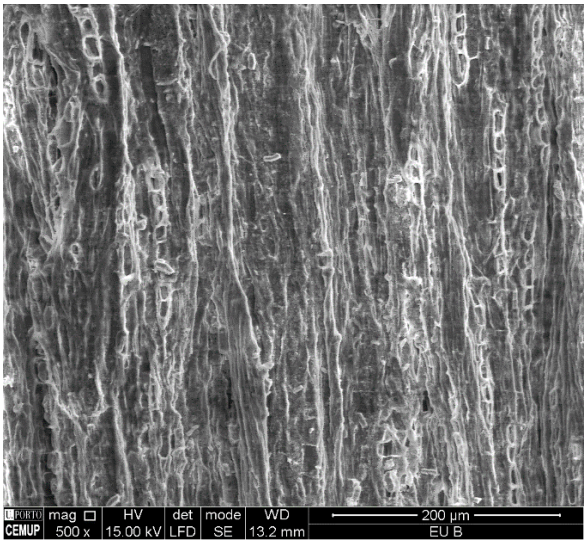


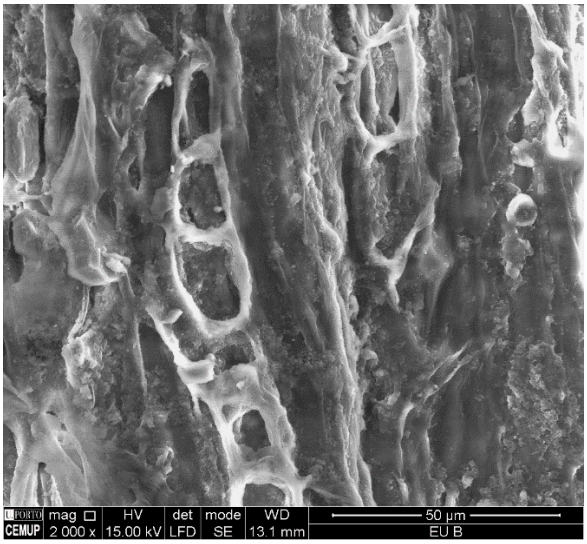
(d)

8.4. Annex 4 SEM images of *Eucalyptus globulus'* Bark

SEM images of *Eucalyptus globulus'* Bark. Scale: (a) 1mm; (b) 200 µm; (c) 50 µm

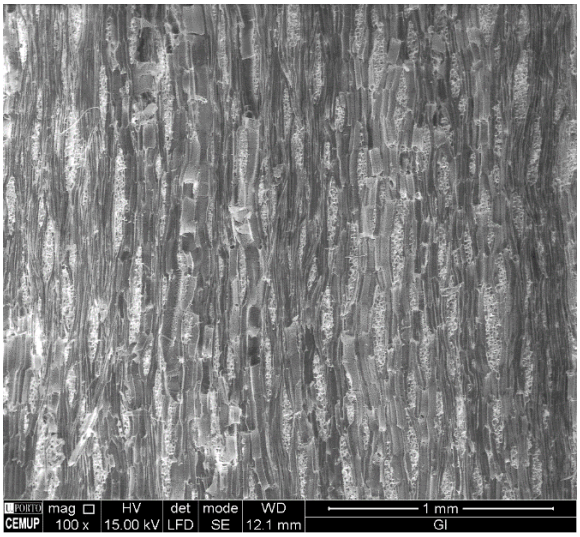


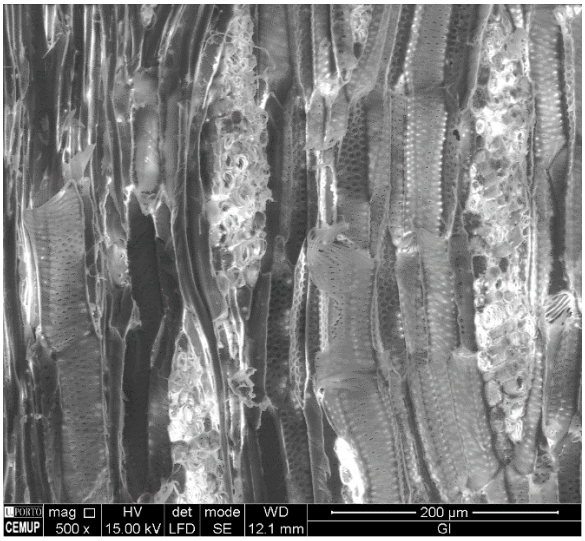


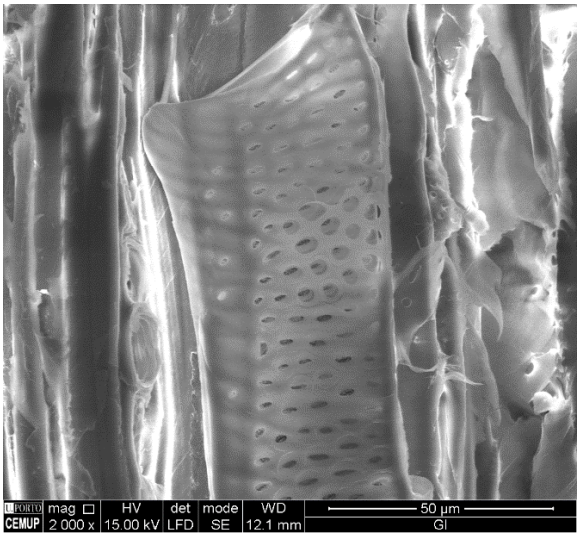


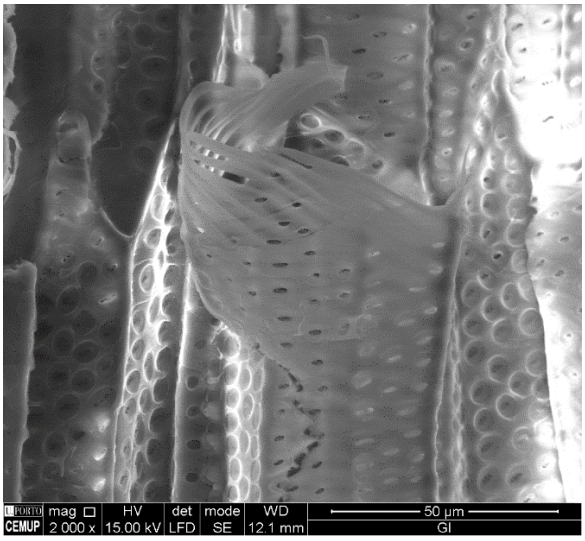
8.5. Annex 5 SEM images of *Genista*

SEM images of *Giniesta*. Scale: (a) 1mm; (b) 200 µm; (c) 50 µm; (d) 50 µm



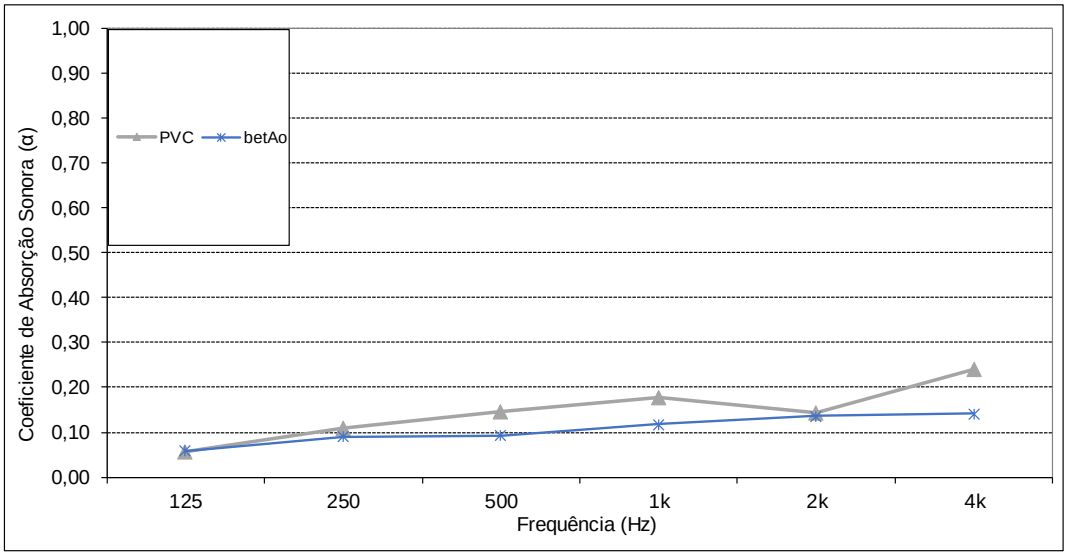


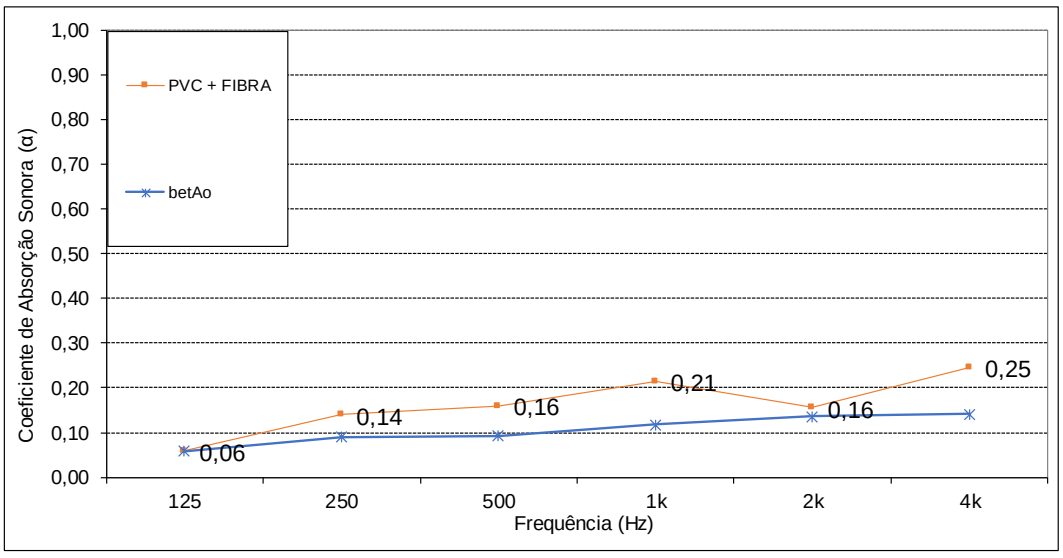




8.6. Annex 6 Concrete sound absorption coefficients

Concrete sample and its sound absorption coefficients compared to RPVC and RPVC_25LCEL

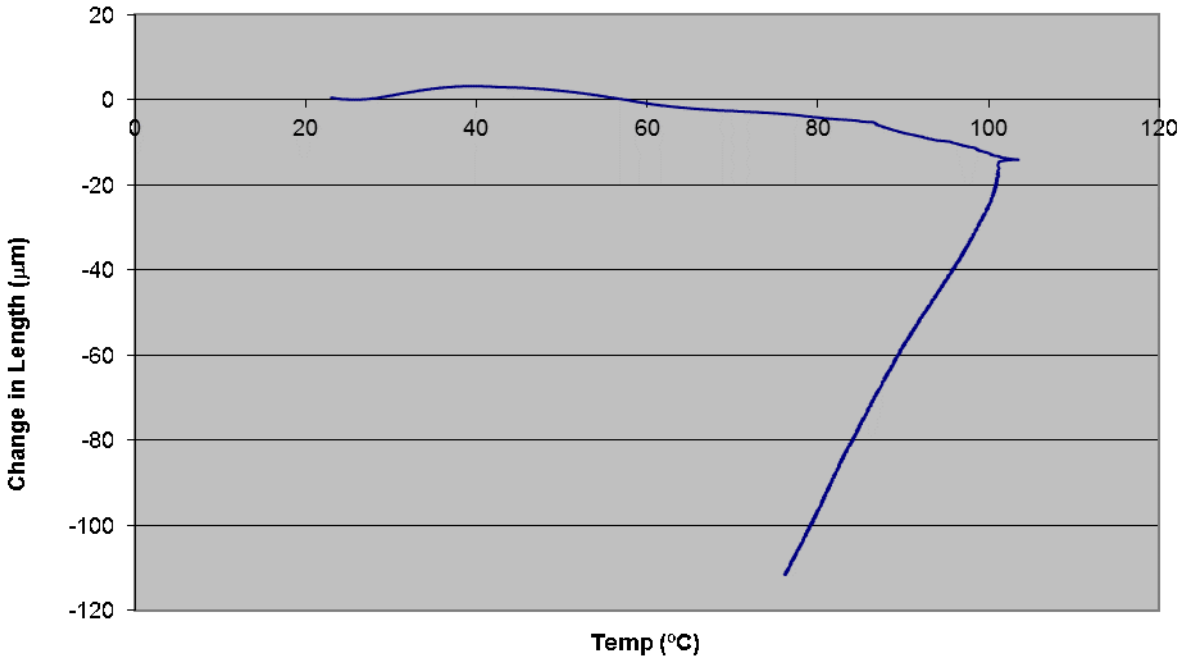




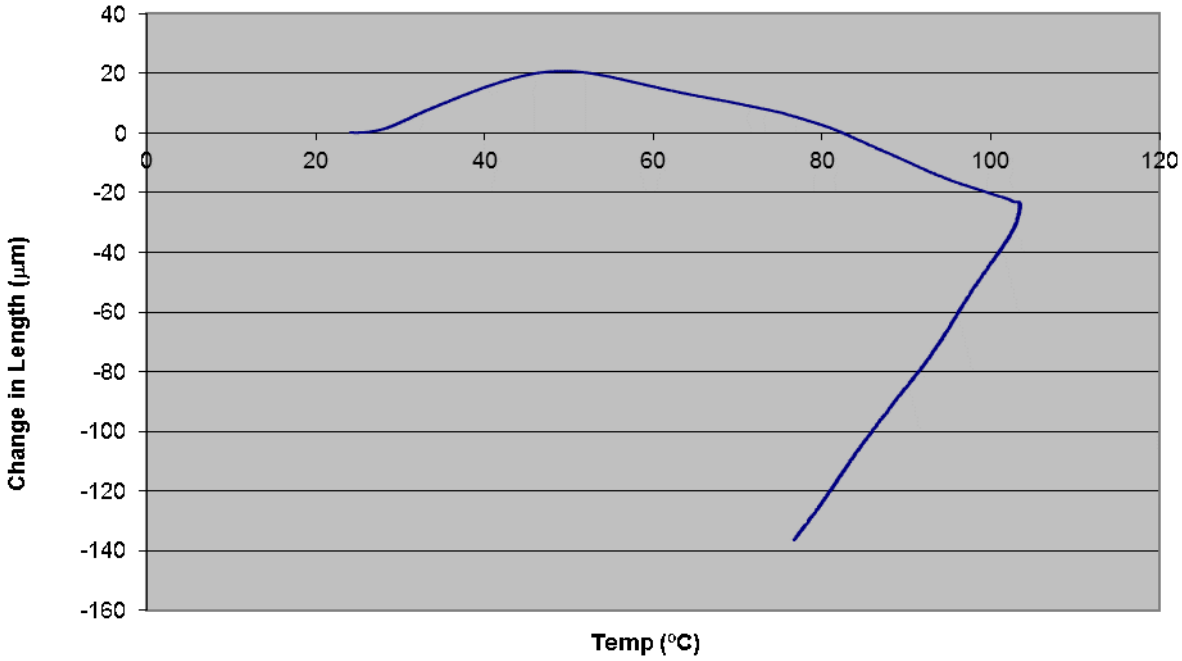
8.7. Annex 7 Dilatometry results

Originally results for dilatometry tests

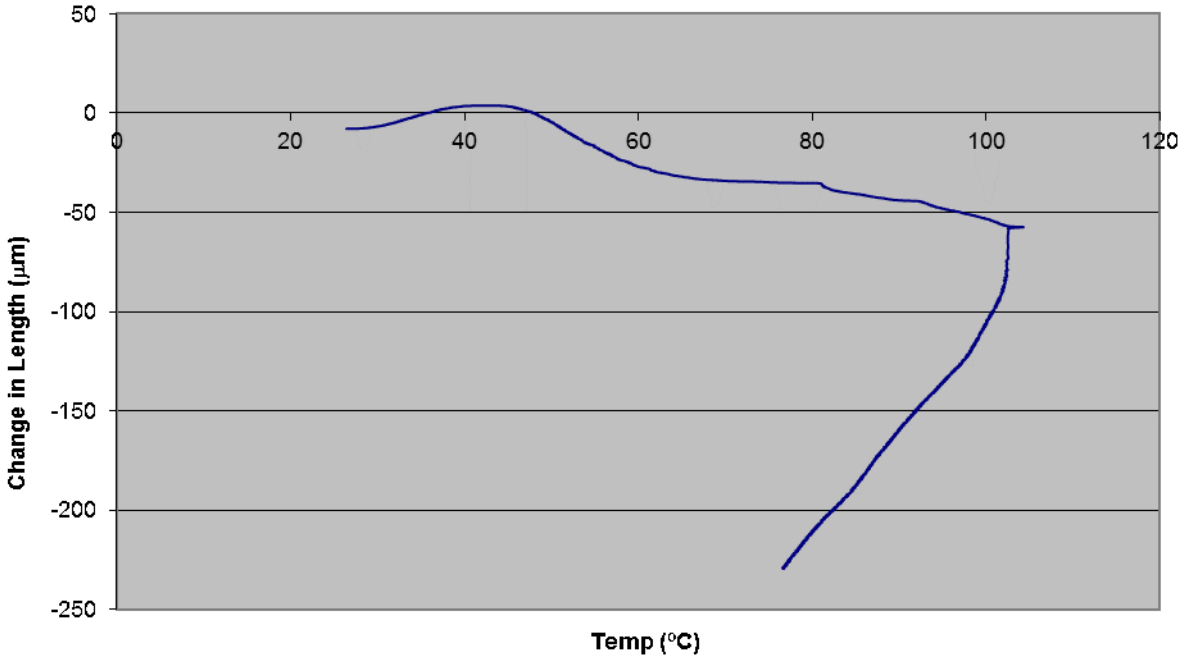
RPVC_25LCEL S#1



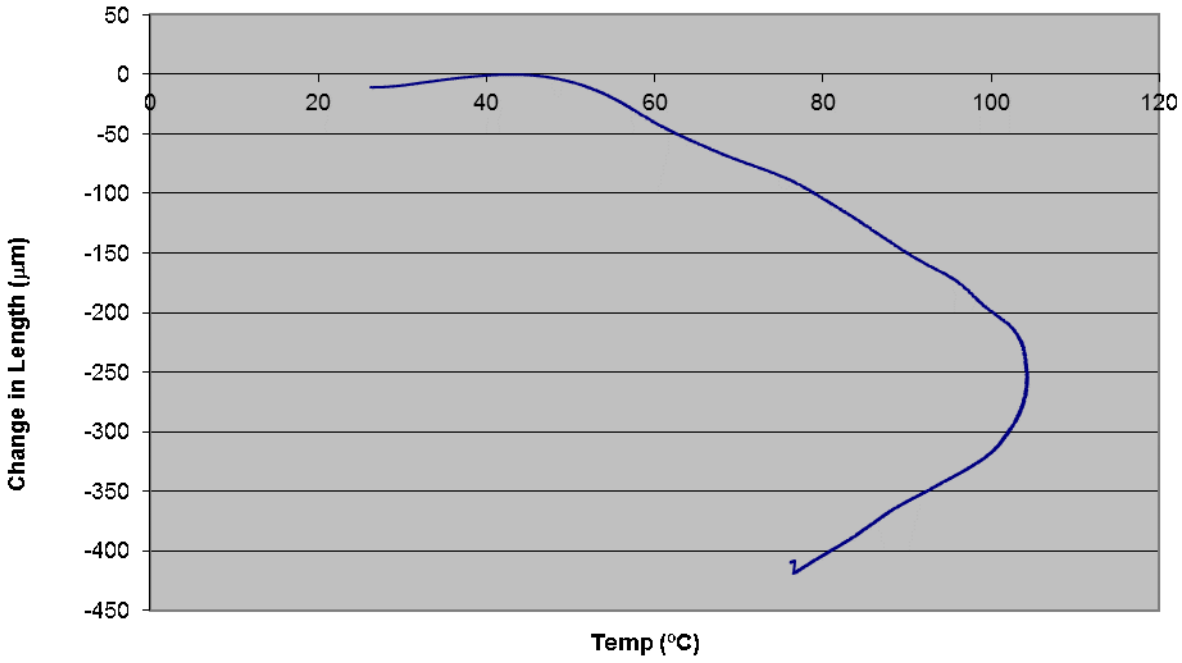
RPVC_25LCEL S#2



RPVC S#1



RPVC S#2



8.8. Annex 8 Norms

On the next pages there are the following norms:

ISO 604

ISO 5274

ISO 14125_1998

INTERNATIONAL STANDARD

ISO 604

Second edition
1993-06-15

Plastics — Determination of compressive properties

Plastiques — Détermination des propriétés en compression

Reference number
ISO 604:1993(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 604 was prepared by Technical Committee ISO/TC 61, *Plastics*, Sub-Committee SC 2, *Mechanical properties*.

This second edition cancels and replaces the first edition (ISO 604:1973), which has been improved with respect to the following points:

— introduction of the compressive modulus;

— simplification with respect to the buckling limit;

— introduction of preferred specimen types, which relate to the multipurpose test specimen according to ISO 3167;

— introduction of three preferred testing speeds, for measuring the modulus and for testing brittle and tough materials respectively.

Annex A forms an integral part of this International Standard. Annex B is for information only.

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland
Printed in Switzerland

Plastics — Determination of compressive properties

1 Scope

1.1 This International Standard specifies a method for determining the compressive properties of plastics under defined conditions. A standard test specimen is defined and its length is adjusted to prevent buckling under load from affecting the results. A range of testing speeds is included.

1.2 The method is used to investigate the compressive behaviour of the test specimens and for determining the compressive strength, compressive modulus and other aspects of the compressive stress/strain relationship under the conditions defined.

1.3 The method applies to the following range of materials:

— rigid and semirigid thermoplastics moulding and extrusion materials, including compounds filled and reinforced by e.g. short fibres, small rods, plates or granules in addition to unfilled types; rigid and semirigid thermoplastic sheet;

— rigid and semirigid thermoset moulding materials, including filled and reinforced compounds; rigid and semirigid thermoset sheet;

— thermotropic liquid crystal polymers.

The method is not normally suitable for use with materials reinforced by textile fibres, rigid cellular materials and sandwich structures containing cellular material.

1.4 The method is performed using specimens which may be either moulded to the chosen dimensions, machined from the central portion of the standard multipurpose test specimen (see ISO 3167) or machined from finished and semifinished products such as mouldings, laminates and extruded or cast sheet.

1.5 The method specifies preferred dimensions for the test specimen. Tests which are carried out on specimens of different dimensions, or on specimens which are prepared under different conditions, may produce results which are not comparable. Other factors, such as the speed of testing and the conditioning of the specimens, can also influence the results. Consequently, when comparative data are required, these factors should be carefully controlled and recorded.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 291:1977, *Plastics — Standard atmospheres for conditioning and testing.*

ISO 293:1986, *Plastics — Compression moulding test specimens of thermoplastic materials.*

ISO 294:—[1], *Plastics — Injection moulding of test specimens of thermoplastic materials.*

ISO 295:1991, *Plastics — Compression moulding of test specimens of thermosetting materials.*

ISO 472:1988, *Plastics — Vocabulary.*

ISO 1268:1974, *Plastics — Preparation of glass fibre reinforced, resin bonded, low-pressure laminated plates or panels for test purposes.*

1) To be published. (Revision of ISO 294:1975)

ISO 2602:1980, *Statistical interpretation of test results — Estimation of the mean — Confidence interval.*

ISO 2818:—[2], *Plastics — Preparation of test specimens by machining.*

ISO 3167:1993, *Plastics — Multipurpose test specimens.*

ISO 5893:1985, *Rubber and plastics test equipment — Tensile, flexural and compression types (constant rate of traverse) — Description.*

3 Principle

The test specimen is compressed along its major axis at constant speed until the specimen fractures or until the load or the decrease in length reaches a predetermined value. The load sustained by the specimen is measured during this procedure.

4 Definitions

For the purposes of this International Standard, the following definitions apply (see also figure 1):

4.1 gauge length, L_0: Initial distance between the gauge marks on the test specimen.

It is expressed in millimetres (mm).

4.2 speed of testing, v: Rate of approach of the plates of the testing machine during the test.

It is expressed in millimetres per minute (mm/min).

4.3 compressive stress, σ (engineering): Compressive load, per unit area of original cross-section, carried by the test specimen (see note 3).

It is expressed in megapascals (MPa).

4.3.1 compressive stress at yield, σ_y: First stress at which an increase in strain (see 4.4) occurs without an increase in stress; may be less than the maximum attainable stress (see figure 1, curve a, and note 3).

4.3.2 compressive strength, σ_M: Maximum compressive stress sustained by the test specimen during a compressive test (see figure 1 and note 3).

4.3.3 compressive stress at break (rupture), σ_B: Compressive stress at break of the test specimen (see figure 1 and note 3).

4.3.4 compressive stress at x % strain, σ_x: Stress at which the strain reaches a specified value of x % (see 4.5).

The compressive stress at x % strain may be measured, e.g., if the stress/strain curve does not exhibit a yield point (see figure 1, curve b, and note 3). In this case, x shall be taken from the relevant product standard or agreed upon by the interested parties. However, in any case, x must be lower than the strain at compressive strength.

4.4 compressive strain, ε: Decrease in length per unit original length of the gauge L_0 [see 8.2, equation (3) and note 3].

It is expressed as a dimensionless ratio or percentage (%).

4.5 nominal compressive strain, ε_c: Decrease in length per unit original length l of the test specimen [see 8.2, equation (4)].

It is expressed as a dimensionless ratio and may be specified directly or as a percentage of the initial length.

4.5.1 nominal compressive yield strain, ε_{cy}: Strain corresponding to the compressive yield stress σ_y (see 4.3.1).

4.5.2 nominal compressive strain at compressive strength, ε_{cM}: Strain corresponding to the compressive strength σ_M (see 4.3.2).

4.5.3 nominal compressive strain at break, ε_{cB}: Strain at break of the test specimen.

4.6 compressive modulus, E_c: Ratio of the stress difference $(\sigma_2 - \sigma_1)$ to the corresponding strain difference values ($\varepsilon_2 = 0{,}002\ 5$ minus $\varepsilon_1 = 0{,}000\ 5$) [see 8.3, equation (7)].

It is expressed in megapascals, MPa.

NOTES

1 The compression modulus is calculated on the basis of the compressive strain ε only (see 4.4).

2 With computer-aided equipment, the determination of the modulus E_c using two distinct stress/strain points may be replaced by a linear regression procedure applied on the part of the curve between these mentioned points.

3 In compression tests the stresses σ and strains ε are negative. The negative sign, however, is generally omitted. If this generates confusion, e.g. in comparing tensile and compressive properties, the negative sign may be added for the latter. This unnecessary for the nominal compressive strains ε_c.

2) To be published. (Revision of ISO 2818:1980)

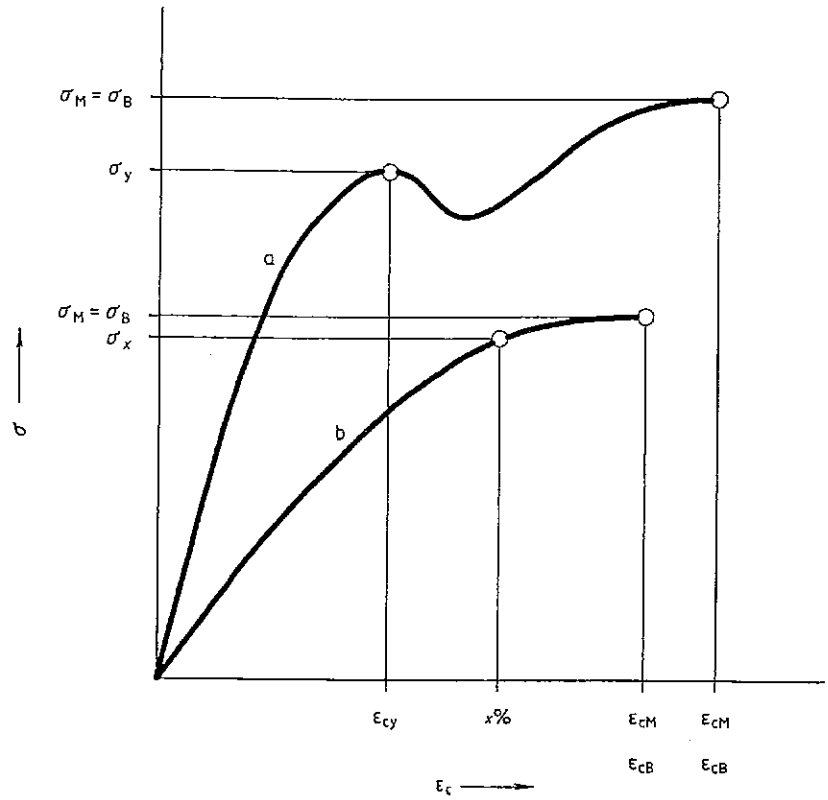


Figure 1 — Typical stress/strain curves

5 Apparatus

5.1 Testing machine

The testing machine shall be power-driven and capable of maintaining the appropriate speed of testing as specified in 7.5. The machine shall satisfy the conditions given in ISO 5893. The testing machine shall be equipped with the devices described in 5.1.1 to 5.1.3.

5.1.1 Compression tool, of hardened steel compression plates, for applying the deformation to the test specimen, so constructed that the load carried by the specimen is axial within 1:1 000 and transmitted through polished surfaces which are flat within 0,025 mm and parallel to each other in a plane normal to the loading axis.

NOTE 4 A self-aligning device may be used where required.

5.1.2 Load indicator, capable of showing the total compressive load carried by the test specimen. The mechanism shall be essentially free of inertia lag at the specified testing speed and shall indicate the load value with an accuracy of ± 1 % or better of the relevant value.

5.1.3 Deformation indicator, suitable for determining the change in length of the appropriate part of the test specimen. If compressive strain ε is to be measured (preferred), then this length is the gauge length; otherwise, for nominal compressive strain ε_c, it is the distance between the contact surfaces of the compression tool. It is desirable, but not essential, that this instrument automatically records this distance. This instrument shall be essentially free of inertia lag at the specified testing speed and shall be accurate to ± 1 % or better of the relevant value.

When a deformation indicator is attached to the test specimen, care shall be taken to ensure that any distortion of or damage to the test specimen is minimal. It is also essential that there is no slippage between the deformation indicator and the test specimen.

5.2 Devices for measuring the dimensions of the test specimens

5.2.1 For rigid materials, use a micrometer or equivalent, reading to at least 0,01 mm, for measuring the thickness, width and length.

5.2.2 For semirigid materials, use a micrometer or equivalent, reading to at least 0,01 mm and provided with a flat circular foot which applies a pressure of 20 kPa ± 3 kPa, for measuring thickness.

6 Test specimens

6.1 Preparation

Prepare test specimens in accordance with the requirements of the International Standard for the material concerned. In the absence of such requirements, the most appropriate method taken from the list of International Standards in clause 2 shall be used, unless otherwise agreed by the interested parties.

All surfaces of the test specimens shall be free from visible flaws, scratches and other imperfections that are likely to influence the results.

6.2 Shape

The test specimen shall be in the shape of a right prism, cylinder or tube. All machining operations shall be carried out carefully so that smooth surfaces result. Great care shall be taken in machining the ends so that smooth, flat, parallel surfaces and sharp, clean edges, to within 0,025 mm perpendicular to the longest axis of the specimen, result.

It is recommended to machine the end surfaces of the test specimen with a lathe or a milling machine.

The dimensions of the test specimens shall meet the conditions in equation (1) (see annex B).

$$\varepsilon_c^* \leqslant 0{,}4\left(\frac{x}{l}\right)^2 \qquad \ldots (1)$$

where

ε_c^* is the maximum nominal compressive strain, expressed as a dimensionless ratio, which occurs during the test;

l is the length of the specimen, measured parallel to the axis of the compressive force;

x is the diameter of the cylinder, the outer diameter of the tube or the thickness (the smaller side of the cross-section) of the prism, depending on the shape of the test specimen.

NOTES

5 For measurement of the compressive modulus E_c according to 4.6, the dimension ratio $x/l \geqslant 0{,}08$ is recommended.

6 When carrying out compression tests in general, the dimension ratio $x/l \geqslant 0{,}4$ is recommended. This corresponds to a maximum compressive strain of about 6 %.

7 Equation (1) is based upon the linear stress/strain behaviour of the material under test. Values of ε_c^* two to three times higher than the maximum strain used in the test should be chosen with increasing compressive strain and ductility of the material.

6.3 Preferred test specimens

The preferred dimensions for test specimens are given in table 1.

Table 1 — Dimensions of preferred specimen types

Dimensions in millimetres

Type	Measurement	Length, l	Width, b	Thickness, h
A	modulus	50 ± 2	10 ± 0,2	4,0 ± 0,2
B	strength	$10\ ^{+0}_{-2}$		

Preferably the specimens are to be cut from a multipurpose test specimen (see ISO 3167).

NOTE 8 Annex A details two types of small test specimen for use when, for reasons of lack of material or geometric constraints for a product, the preferred specimens cannot be used.

6.4 Gauge marks

If optical deformation indicators are used, it is necessary to put gauge marks on the specimen to define the gauge length. These shall be approximately equidistant from the midpoint of the test specimen, and the distance between the marks shall be measured to an accuracy of 1 % or better.

Gauge marks shall not be scratched, punched or impressed upon the test specimen in any way which causes damage to the material being tested. It must be ensured that the marking medium has no detrimental effect on the material being tested and that, in the case of two pairs of parallel lines, they are as narrow as possible.

6.5 Anisotropic materials

6.5.1 In the case of anisotropic materials, the test specimens shall be chosen so that the compressive stress in the test procedure will be applied in the same or similar direction to that experienced by the products (moulded articles, sheet, tubes, etc.) during their application in service, if known.

The relationship between the dimensions of the test specimen and the size of the product will determine the possibility of using preferred test specimens. If the use of the preferred test specimen is impossible, the size of the product will govern the choice of dimensions of the test specimens in accordance with

6.2 as well. It should be noted that the orientation and the dimensions of the test specimens sometimes have a very significant influence on the test results. This is particularly true of laminates.

6.5.2 When the material shows a significant difference in compressive properties in two principal directions, it shall be tested in these two directions. If, because of its destined application, this material will be subjected to compressive stress at some specific orientation to the principal direction, it is desirable to test the material in that orientation.

The orientation of the test specimens relative to the principal directions shall be recorded.

6.6 Number of test specimens

6.6.1 Test at least five specimens for each sample in the case of isotropic materials.

6.6.2 Test at least ten specimens, five normal to, and five parallel to the principal axis of anisotropy for each sample, in the case of anisotropic materials.

6.6.3 Specimens that break at some obvious flaw shall be discarded and replacement specimens shall be tested.

6.7 Conditioning of test specimens

The test specimens shall be conditioned in accordance with the requirements of the International Standard for the material. In the absence of such requirements, use shall be made of the most appropriate conditions given in ISO 291, unless otherwise agreed by the interested parties.

The preferred condition is atmosphere 23/50, except when the compressive properties of the material are known to be insensitive to moisture, in which case humidity control is unnecessary.

7 Test procedure

7.1 Perform the test in one of the standard atmospheres specified in ISO 291, preferably the same atmosphere as used for conditioning.

7.2 Measure the width and thickness, or the diameter(s), of the test specimen at three points along its length and calculate the mean value of the cross-sectional area.

Measure the length of each test specimen, to 1 % accuracy.

7.3 Place the test specimen between the surfaces of the compression plates and align the centreline of the compression plate surfaces. Ensure that the end surfaces of the specimen are parallel to the surfaces of the compression plates and adjust the machine so that the surfaces of the ends of the test specimen and compression plate are just touching.

NOTE 9 During compression, the end surfaces of the test specimen may slip along the compression plates to varying extents, depending upon the surface textures of the specimen and plates. This will lead to varying degrees of barrel distortion, which in turn may influence the properties to be measured. The less rigid the material, the more pronounced the effect.

For the most precise measurements, it is recommended that either the end surfaces be treated with an appropriate lubricant to promote slip or that discs of fine abrasive paper be used between specimen and plates to inhibit slip. If either method is used, it shall be noted in the test report.

7.4 Attach the deformation indicator, if required.

7.5 Set the speed of testing v in millimetres per minute (see 4.2) in accordance with the material specification or, in the absence of this, to that of the following value:

$1 \pm 0{,}2$

$2 \pm 0{,}4$

5 ± 1

10 ± 2

20 ± 2

which is the closest approximation to

$v = 0{,}02l$ (l in millimetres)	for modulus measurements;
$v = 0{,}1l$ (l in millimetres)	for strength measurements with brittle materials, which break prior to yielding;
$v = 0{,}5l$ (l in millimetres)	for strength measurements with ductile materials, which yield.

For the preferred test specimens (see 6.3) the testing speeds are

1 mm/min for modulus measurements (l = 50 mm);

1 mm/min for strength measurements with brittle materials (l = 10 mm);

5 mm/min for strength measurements with ductile materials (l = 10 mm).

7.6 Determine the force (stress) and the corresponding compression (strain) of the specimen during the test. It is preferable to use an automatic recording system, which yields a complete stress/strain curve, for this operation.

7.7 Determine all relevant stresses and strains compiled in clause 4 (definitions) from the stress/strain curve or by other suitable means.

7.8 The modulus, as defined in 4.6, may be determined from the stress/strain curve, provided that the stress and strain scales are sufficiently expanded.

8 Calculation and expression of results

8.1 Stress calculations

Calculate all stress values defined in 4.3 on the basis of the original cross-sectional area of the test specimen:

$$\sigma = \frac{F}{A} \qquad \ldots (2)$$

where

σ is the compressive stress value in question, in megapascals;

F is the measured force in question, in newtons;

A is the initial mean cross-sectional area of the specimen, in square millimetres.

8.2 Strain calculations

Calculate the compressive strain defined in 4.4 on the basis of the gauge length defined in 4.1 using the equations:

$$\varepsilon = \frac{\Delta L}{L_0} \qquad \ldots (3)$$

$$\varepsilon\,(\%) = 100 \times \frac{\Delta L}{L_0} \qquad \ldots (4)$$

The nominal compressive strain, defined in 4.5, is calculated on the basis of the initial specimen length l using the equations:

$$\varepsilon_c = \frac{\Delta l}{l} \qquad \ldots (5)$$

$$\varepsilon_c\,(\%) = 100 \times \frac{\Delta l}{l} \qquad \ldots (6)$$

where

ε is the compressive strain, expressed as a dimensionless ratio or in percent;

ε_c is the nominal compressive strain, expressed as a dimensionless ratio or in percent;

L_0 is the initial distance between the gauge marks (gauge length) on the test specimen, expressed in millimetres;

ΔL is the decrease in the specimen length between the gauge marks, expressed in millimetres;

l is the initial specimen length, expressed in millimetres;

Δl is the decrease in the specimen length, expressed in millimetres.

8.3 Modulus calculation

Calculate the compressive modulus, defined in 4.6, using equation (7):

$$E_c = \frac{\sigma_2 - \sigma_1}{\varepsilon_2 - \varepsilon_1} \qquad \ldots (7)$$

where

E_c is the compressive modulus of elasticity, expressed in megapascals;

σ_1 is the compressive stress calculated according to equation (2), in megapascals, measured at the strain value ε_1;

σ_2 is the compressive stress calculated according to equation (2), in megapascals, measured at the strain value ε_2;

ε_1 is the compressive strain calculated according to equation (3) or (4), having the value $\varepsilon_1 = 0{,}000\ 5$ or 0,05 %;

ε_2 is the compressive strain calculated according to equation (3) or (4), having the value $\varepsilon_2 = 0{,}002\ 5$ or 0,25 %.

8.4 Statistical parameters

Calculate the arithmetic mean of each five test results and, if required, the standard deviation and 95 % confidence interval of the mean value by the procedure given in ISO 2602.

8.5 Significant figures

Calculate the compressive stress and modulus to three significant figures. Calculate the compressive strain to two significant figures.

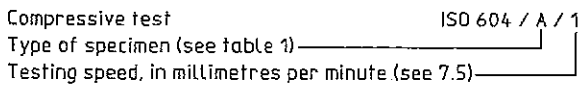
9 Precision

The precision of this test method is not known because interlaboratory data are not available. When interlaboratory data are obtained, a precision statement will be added with the next revision.

10 Test report

The test report shall include the following information:

a) a reference to this International Standard, including the type of specimen and the testing speed according to



b) complete identification of the material tested, including type, source, manufacturer's code number and history, where these are known;

c) a description of the nature and form of the material in terms of whether it is a product, semifinished product, test plate or specimen. It should include principal dimensions, shape, method of manufacture, order of layers, preliminary treatments, etc.;

d) type of test specimen, width, thickness and length: mean, minimum and maximum values, if applicable;

e) method of preparing the test specimen and any details of the manufacturing method used;

f) if the material is in the form of a product or a semifinished product, the orientation of the specimen in relation to the product or semifinished product from which it is cut;

g) number of specimens tested;

h) the standard atmosphere for conditioning and for testing, plus any special conditioning treatment, if required by the International Standard for the material or product;

i) accuracy grading of the test machine (see ISO 5893);

j) type of deformation indicator;

k) type of compression tool;

l) whether or not slip promoters or slip inhibitors were used on the end surfaces;

m) the speed of testing;

n) the individual test results;

o) the mean value(s) x of the measured property(ies), quoted as the indicative value(s) for the material tested;

p) (optionally) the standard deviation SD, and/or coefficient of variation, and/or confidence limits of the mean;

q) if any test specimens have been rejected and replaced, and if so, the reasons;

r) date of measurement.

Annex A
(normative)

Small test specimens

A.1 Test specimens as defined in clause 6 may be impossible to produce from the amount of material available or from a finished product.

In these circumstances, use may be made of the small specimens described in this annex.

A.2 It must be expected that the results obtained with small specimens will differ from those obtained with normal-sized specimens.

A.3 The use of small specimens shall be agreed to by the interested parties, and specific reference to their use made in the test report.

A.4 The test shall be carried out in accordance with this International Standard for normal test specimens, except as noted below.

The nominal dimensions of the specimens, in millimetres, shall be as specified in table A.1.

Table A.1 — Nominal dimensions of small test specimens

Dimensions in millimetres

Dimension	Type 1	Type 2
Thickness	3	3
Width	5	5
Length	6	35

The type 2 specimen shall only be used for determining compressive modulus; in this case the use of a 15 mm gauge length is recommended to facilitate the measurement.

Annex B
(informative)

Limit of buckling

According to Euler, the critical axial compressive force, F^*, for the onset of buckling of a specimen fixed at both ends, calculated for linear stress-strain behaviour of the material under test is

$$F^* = \frac{\pi^2 E_c I}{l^2} \quad \ldots (B.1)$$

where I is the least second moment of the cross-sectional area.

The critical force can be replaced by the corresponding nominal strain at buckling in accordance with equation (B.2):

$$F^* = E_c \cdot A \cdot \varepsilon_b \quad \ldots (B.2)$$

where

A is the cross-sectional area;

ε_b is the nominal compressive strain at buckling.

This gives the critical buckling strain, which depends only upon the dimensions of the specimen, in accordance with equation (B.3):

$$\varepsilon_b = \pi^2 \times \frac{I}{Al^2} \quad \ldots (B.3)$$

For the different types of specimen shape, equation (B.3) can be expressed as follows:

a) For a right prism

$$\varepsilon_b = \frac{\pi^2}{12}\left(\frac{h}{l}\right)^2 \quad \ldots (B.4)$$

b) For a right cylinder or tube

$$\varepsilon_b = \frac{\pi^2}{16}\left(\frac{2r}{l}\right)^2\left[1+\left(\frac{r_1}{r}\right)^2\right] \quad \ldots (B.5)$$

where

l is the length of the right prism, cylinder or tube, i.e. the dimension parallel to the compressive force;

h is the thickness of the right prism, i.e. the smaller side of the cross-section;

r is the radius of the cylinder or the outer radius of the tube;

r_1 is the inner radius of the tube (zero for a cylinder).

Compared to the cylinder, the additional stability of the tube in accordance with equation (B.5) cannot be used, as thin-walled tubes fail in accordance with additional buckling modes not discussed here. The numerical factors used in equations (B.4) and (B.5) equal 0,8 and 0,6 respectively. As these equations give only a rough estimate of the strain of buckling, they can be approximated to the general equation (1) in 6.2, in which the numerical factor chosen has been decreased to avoid buckling.

In the first edition of this International Standard (ISO 604:1973), the "slenderness ratio" λ, where

$$\lambda = l\sqrt{\frac{A}{I}}$$

was used with a recommended value of 10. As can be seen from equation (B.3) this corresponds to a buckling strain ε_b of 10 %. The magnitude of ε_b, however, is a direct measure of the test range which can be used with the specimen under test. The "least radius of gyration" i, where

$$i = \sqrt{\frac{I}{A}}$$

was also used in the first edition of this International Standard. Neither λ nor i are used in the present second edition.

UDC [678.5/.8].017:539.411

Descriptors: plastics, tests, compression tests, determination, compressibility.

Price based on 9 pages

Determination of tensile properties of plastics

Part 4: Test conditions for isotropic and orthotropic fibre-reinforced plastic composites (ISO 527-4:1997)

English version of DIN EN ISO 527-4

DIN EN ISO 527-4

ICS 83.120

Supersedes DIN EN 61, November 1977 edition.

Descriptors: Plastics, composites, testing, tensile strength.

Kunststoffe – Bestimmung der Zugeigenschaften – Teil 4: Prüfbedingungen für isotrop und anisotrop faserverstärkte Kunststoffverbundwerkstoffe (ISO 527-4:1997)

European Standard EN ISO 527-4:1997 has the status of a DIN Standard.

A comma is used as the decimal marker.

National foreword

This standard has been published in accordance with a decision taken by CEN/TC 249 to adopt, without alteration, International Standard ISO 527-4 as a European Standard.

The responsible German body involved in its preparation was the *Normenausschuß Kunststoffe* (Plastics Standards Committee), Technical Committee *Prepregs.*

DIN EN ISO 527-1 and DIN EN ISO 527-2 are the standards corresponding to International Standards ISO 527-1 and ISO 527-2, respectively, referred to in clause 2 of the EN.

Amendments

DIN EN 61, November 1977 edition, has been superseded by the specifications of EN ISO 527-4, which is identical to ISO 527-4.

Previous edition

DIN EN 61: 1977-11.

National Annex NA

Standards referred to

(and not included in **Normative references** and **Annex ZA**)

DIN EN ISO 527-1 Plastics – Determination of tensile properties – Part 1: General principles (ISO 527-1:1993 including Corr. 1:1994)

DIN EN ISO 527-2 Plastics – Determination of tensile properties – Part 2: Test conditions for moulding and extrusion plastics

EN comprises 12 pages.

Ref. No. DIN EN ISO 527-4:1997-07
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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN ISO 527-4

April 1997

ICS 83.120

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English version

Plastics

Determination of tensile properties

Part 4: Test conditions for isotropic and orthotropic fibre-reinforced plastic composites

(ISO 527-4:1997)

Plastiques – Détermination des propriétés en traction – Partie 4: Conditions d'essai pour les composites plastiques renforcés de fibres isotropes et orthotropes
(ISO 527-4:1997)

Kunststoffe – Bestimmung der Zugeigenschaften – Teil 4: Prüfbedingungen für isotrop und anisotrop faserverstärkte Kunststoffverbundwerkstoffe
(ISO 527-4:1997)

This European Standard was approved by CEN on 1997-03-28.

CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart 36, B-1050 Brussels

Ref. No. EN ISO 527-4:1997 E

Foreword

International Standard

ISO 527-4:1997 Plastics – Determination of tensile properties – Part 4: Test conditions for isotropic and orthotropic fibre-reinforced plastic composites,

which was prepared by ISO/TC 61 'Plastics' of the International Organization for Standardization, has been adopted by Technical Committee CEN/TC 249 'Plastics', the Secretariat of which is held by IBN, as a European Standard.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, and conflicting national standards withdrawn, by October 1997 at the latest.

In accordance with the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard:

Austria, Belgium, the Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, the Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 527-4:1997 was approved by CEN as a European Standard without any modification.

NOTE: Normative references to international publications are listed in Annex ZA (normative).

1 Scope

1.1 This part of ISO 527 specifies the test conditions for the determination of the tensile properties of isotropic and orthotropic fibre-reinforced plastic composites, based upon the general principles given in part 1.

Unidirectionally reinforced materials are covered by part 5.

1.2 See ISO 527-1, subclause 1.2.

1.3 The test method is suitable for use with the following materials:

— fibre-reinforced thermosetting and thermoplastic composites incorporating non-unidirectional reinforcements such as mats, woven fabrics, woven rovings, chopped strands, combinations of such reinforcements, hybrids, rovings, short or milled fibres or preimpregnated materials (prepregs) (for directly injection-moulded specimens, see specimen 1A in ISO 527-2:1993);

— combinations of the above with unidirectional reinforcements and multidirectional reinforced materials constructed from unidirectional layers, provided such laminates are symmetrical (for materials with completely, or mainly, unidirectional reinforcements, see ISO 527-5);

— finished products made from these materials.

The reinforcement fibres covered include glass fibres, carbon fibres, aramid fibres and other similar fibres.

1.4 The method is performed using specimens machined from a test panel made in accordance with ISO 1268 or by equivalent methods, or from finished and semi-finished products with suitable flat areas.

1.5 See ISO 527-1, subclause 1.5.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 527. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 527 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 527-1:1993, *Plastics — Determination of tensile properties — Part 1: General principles.*

ISO 527-2:1993, *Plastics — Determination of tensile properties — Part 2: Test conditions for moulding and extrusion plastics.*

ISO 527-5:1997, *Plastics — Determination of tensile properties — Part 5: Test conditions for unidirectional fibre-reinforced plastic composites.*

ISO 1268:1974, *Plastics — Preparation of glass fibre reinforced, resin bonded, low-pressure laminated plates or panels for test purposes.*

ISO 2818:1994, *Plastics — Preparation of test specimens by machining.*

ISO 3534-1:1993, *Statistics — Vocabulary and symbols — Part 1: Probability and general statistical terms.*

3 Principle

See ISO 527-1, clause 3.

4 Definitions

For the purposes of this part of ISO 527, the following definitions apply.

4.1 gauge length: See ISO 527-1, subclause 4.1.

4.2 speed of testing: See ISO 527-1, subclause 4.2.

4.3 tensile stress, σ (engineering)**:** See ISO 527-1, subclause 4.3, except that σ for "1"-direction specimens is defined as σ_1 and for "2"-direction specimens as σ_2 (see 4.8 for definitions of these directions).

4.3.1 tensile strength, σ_M**:** See ISO 527-1, subclause 4.3.3, except that σ_M for "1"-direction specimens is defined as σ_{M1} and for "2"-direction specimens as σ_{M2}.

4.4 tensile strain, ε**:** See ISO 527-1, subclause 4.4, except that ε for "1"-direction specimens is defined as ε_1 and for "2"-direction specimens as ε_2.

It is expressed as a dimensionless ratio or in percent.

4.5 tensile strain at tensile strength; tensile failure strain, ε_M**:** The tensile strain at the point corresponding to the tensile strength of the specimen.

For "1"-direction specimens, ε_M is defined as ε_{M1} and for "2"-direction specimens as ε_{M2}.

It is expressed as a dimensionless ratio or in percent.

4.6 modulus of elasticity in tension; Young's modulus, E**:** See ISO 527-1, subclause 4.6, except that E for "1"-direction specimens is defined as E_1 and for "2"-direction specimens as E_2.

The strain values used are as given in ISO 527-1, subclause 4.6, i.e. $\varepsilon' = 0{,}000\ 5$ and $\varepsilon'' = 0{,}002\ 5$ (see figure 1), unless alternative values are given in the material or technical specifications.

4.7 Poisson's ratio, μ**:** See ISO 527-1, subclause 4.7, except that for "1"-direction specimens μ_b is defined as μ_{12} and μ_h as μ_{13}, using the coordinates shown in figure 2. For "2"-direction specimens, μ_b is defined as μ_{21} and μ_h as μ_{23}.

4.8 specimen coordinate axes: The "1"-direction is normally defined in terms of a feature associated with the material structure or the production process, such as the length direction in continuous-sheet processes (see figure 2). The "2"-direction is perpendicular to the "1"-direction.

NOTES

1 The "1"-direction is also referred to as the 0° or longitudinal direction and the "2"-direction as the 90° or transverse direction-

2 For unidirectional materials covered by part 5 of this International Standard, the direction parallel to the fibres is defined as the "1"-direction and the direction perpendicular to the fibres (in the plane of the fibres) as the "2"-direction.

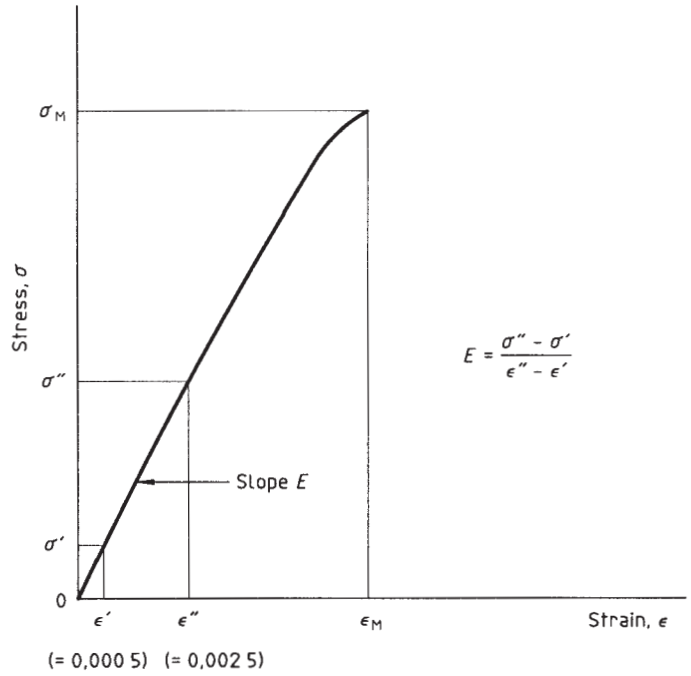


Figure 1 — Stress-strain curve

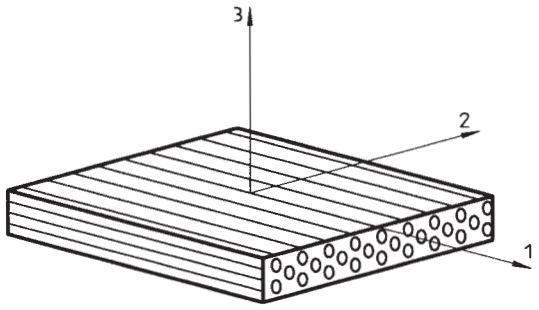


Figure 2 — Fibre-reinforced plastic composite showing axes of symmetry

5 Apparatus

See ISO 527-1, clause 5, except for the following: The micrometer or its equivalent (see 5.2.1) shall read to 0,01 mm or better. It shall have a suitable-size ball-ended anvil if used on irregular surfaces and a flat anvil if used on flat, smooth (e.g. machined) surfaces.

Subclause 5.2.2 does not apply.

NOTE — It is recommended that alignment of the specimen and loading train be checked as described in annex B.

6 Test specimens

6.1 Shape and dimensions

Three types of test specimen are specified for use with this part of ISO 527, as detailed and illustrated in figure 3 (type 1B) and figure 4 (types 2 and 3).

Type 1B is for testing fibre-reinforced thermoplastics. Type 1B specimens may also be used for fibre-reinforced thermosets if they break within the gauge length. Type 1B shall not be used for multidirectional, continuous-fibre-reinforced materials.

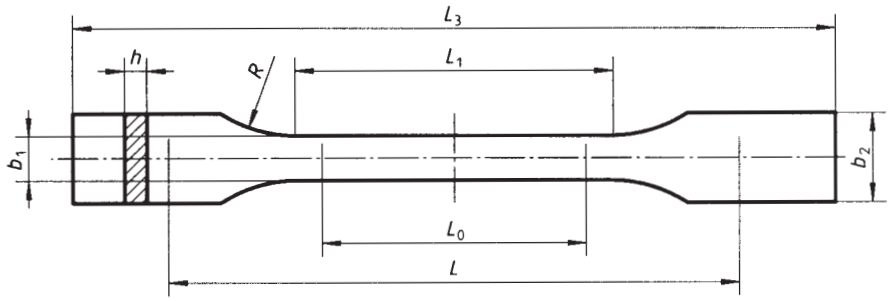
Type 2 (rectangular without end tabs) and type 3 (rectangular with bonded end tabs) are for testing fibre-reinforced thermosets and thermoplastics. Specimens with unbonded end tabs are considered as type 2.

The preferred width of type 2 and type 3 specimens is 25 mm, but widths of 50 mm or greater may be used if the tensile strength is low due to the particular type of reinforcement used.

The thickness of type 2 and type 3 specimens shall be between 2 mm and 10 mm.

To decide whether to use type 2 or type 3 specimens, first carry out tests with type 2 specimens and, if the test is not possible or not satisfactory, i.e. if the specimen slips or breaks in the grips (see ISO 527-1, subclause 5.1), use type 3 specimens.

For compression-moulded materials, the thickness between the end-pieces of any type of specimen shall at no point deviate from the mean by more than 2 %.



Dimensions in millimetres

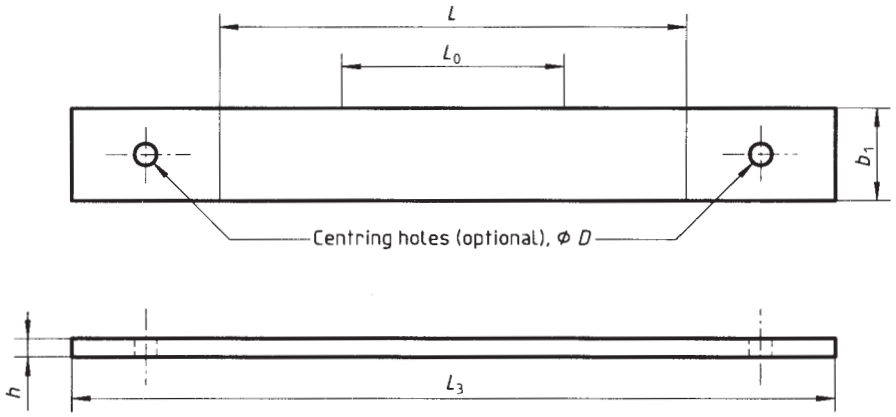
L_3	Overall length	$\geqslant$ 150 [1]
L_1	Length of narrow parallel-sided portion	60 ± 0,5
R	Radius	$\geqslant$ 60 [2]
b_2	Width at ends	20 ± 0,2
b_1	Width of narrow portion	10 ± 0,2
h	Thickness	2 to 10
L_0	Gauge length (recommended for extensometers)	50 ± 0,5
L	Initial distance between grips	115 ± 1

NOTE — Requirements on specimen quality and parallelism are given in clause 6.

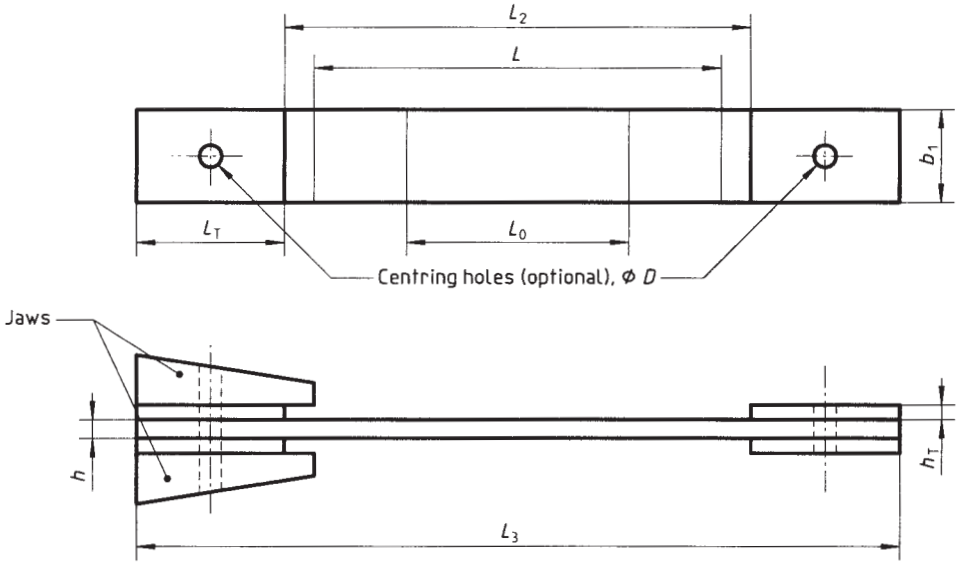
1) For some materials, the length of the tabs may need to be extended (e.g. so that l_3 = 200 mm) to prevent breakage or slippage of the specimen in the jaws.

2) It should be noted that a thickness of 4 mm gives a specimen which is identical to the type 1B specimen specified in ISO 527-2 and ISO 3167:1993, *Plastics — Multipurpose test specimens.*

Figure 3 — Type 1B specimen



a) Type 2 specimen



b) Type 3 specimen

Dimensions in millimetres

		Type 2	Type 3
L_3	Overall length	⩾ 250	⩾ 250
L_2	Distance between end tabs	—	150 ± 1
b_1	Width	25 ± 0,5 or 50 ± 0,5	25 ± 0,5 or 50 ± 0,5
h	Thickness	2 to 10	2 to 10
L_0	Gauge length (recommended for extensometers)	50 ± 1	50 ± 1
L	Initial distance between grips	150 ± 1	136 (nominal)
L_T	Length of end tabs	—	⩾ 50
h_T	Thickness of end tabs	—	1 to 3
D	Diameter of centring holes	3 ± 0,25	3 ± 0,25

NOTE — Requirements on specimen quality and parallelism are given in clause 6.

Figure 4 — Type 2 and type 3 specimens

6.2 Preparation of specimens

6.2.1 General

In the case of moulding and lamination materials, prepare a panel in accordance with ISO 1268 or another specified/agreed procedure. Cut individual specimens, or groups of specimens in the case of type 3 specimens (see annex A), from the panel.

In the case of finished products (for example, for quality control during manufacture or on delivery), take specimens from flat areas.

Parameters for machining specimens are specified in ISO 2818. Further guidance on cutting specimens is given in annex A.

6.2.2 End tabs (for type 3 specimens)

The ends of the specimen shall be reinforced, preferably with end tabs made of cross-ply or fabric glass-fibre/resin laminate with the fibres at ± 45° to the specimen axis. The tab thickness shall be between 1 mm and 3 mm, with a tab angle of 90° (i.e. not tapered).

Alternative tabbing arrangements are permissible, but shall be shown, before use, to give at least equal strength and no greater coefficient of variation (see ISO 527-1, subclause 10.5, and ISO 3534-1) than the recommended tabs. Possible alternatives include tabs made from the material under test, mechanically fastened tabs, unbonded tabs made of rough materials (such as emery paper or sandpaper, and the use of roughened grip faces).

6.2.3 Application of end tabs (for type 3 specimens)

Bond the end tabs to the specimen with a high-stretch adhesive as described in annex A.

NOTE — The same procedure can be used for individual specimens and for a group of specimens.

6.3 Gauge marks

See ISO 527-1, subclause 6.3.

6.4 Checking the specimens

See ISO 527-1, subclause 6.4.

6.5 Anisotropy

The properties of fibre-reinforced plastic composites frequently vary with direction in the plane of the sheet (anisotropy). For this reason, it is recommended that two groups of test specimens be prepared with their major axes parallel and perpendicular, respectively, to the direction of some feature which is inferred from a knowledge of the structure of the material or its method of manufacture (see subclause 4.8).

7 Number of specimens

See ISO 527-1, clause 7.

8 Conditioning

See ISO 527-1, clause 8.

9 Procedure

9.1 Test atmosphere

See ISO 527-1, subclause 9.1.

9.2 Measurement of specimen dimensions

See ISO 527-1, subclause 9.2.

9.3 Clamping

See ISO 527-1, subclause 9.3.

9.4 Prestresses

See ISO 527-1, subclause 9.4.

9.5 Setting of extensometers and strain gauges and placing of gauge marks

See ISO 527-1, subclause 9.5. Measure the gauge length to an accuracy of 1 % or better.

9.6 Test speed

Use the following test speeds:

9.6.1 For type 1B test specimens

a) 10 mm/min for routine quality control;

b) 2 mm/min for qualification tests,

when measuring the maximum elongation,

when determining the tensile modulus of elasticity.

9.6.2 For type 2 and type 3 test specimens

a) 5 mm/min for routine quality control;

b) 2 mm/min for qualification tests,

when measuring the maximum elongation,

when determining the tensile modulus of elasticity.

9.7 Recording of data

See ISO 527-1, subclause 9.7.

10 Calculation and expression of results

See ISO 527-1, clause 10, except that the definitions given in clause 4 of this part of ISO 527 apply and strain values shall be reported to three significant figures.

If Poisson's ratio is required, calculate it at the strain values given in 4.6.

11 Precision

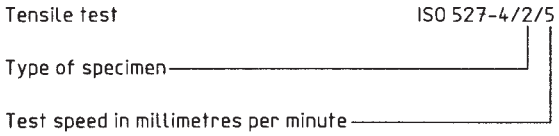
The precision of this test method is not known because interlaboratory data are not available. When interlaboratory data are obtained, a precision statement will be added with the following revision.

The precision data will be specific to particular combinations of fibre and matrix types.

12 Test report

The test report shall include the following information:

a) a reference to this part of ISO 527, including the type of specimen and the test speed, written in the following format:



b) to q) see ISO 527-1, clause 12, b) to q), including fibre type, fibre content and fibre geometry (e.g. mat) in 12b).

Annex A
(normative)

Specimen preparation

A.1 Machining the specimens

In all cases take the following precautions:

— Avoid working under conditions that would create a large build-up of heat in the specimen (the use of a coolant is recommended). If a liquid coolant is used, dry the specimens immediately after machining.

— Check that all cut surfaces of the specimen are free from machining defects.

A.2 Preparation of specimens with bonded end tabs

A recommended method is as follows:

Cut out from the material under test a sheet having the length of the intended specimens and of a width suitable for the number of specimens required.

Identify the "1"-direction of the material in the sheet.

Cut out rectangular strips of the required length and width for the tabs.

Attach the strips to the sheet as follows:

a) If required, rub with fine abrasive paper or blast with suitable sand all the surfaces to which adhesive will be applied.

b) Remove all dust from these surfaces and clean them with a suitable solvent.

c) Bond the strips in place along the ends of the sheet, parallel to each other and normal to the length direction of the specimens, as shown in figure A.1, using a high-stretch adhesive and strictly following the adhesive manufacturer's instructions.

NOTE — It is recommended that a film adhesive with a thin carrier be used. The adhesive should preferably have a shear strength greater than 30 MPa. It is desirable that the adhesive used be flexible in nature, with an elongation at break greater than that of the material under test.

d) Keep the bonded parts at the pressure and temperature recommended by the adhesive manufacturer for the time recommended by the manufacturer.

e) Cut the sheet, together with the strips constituting the end tabs, into test specimens (see figure A.1).

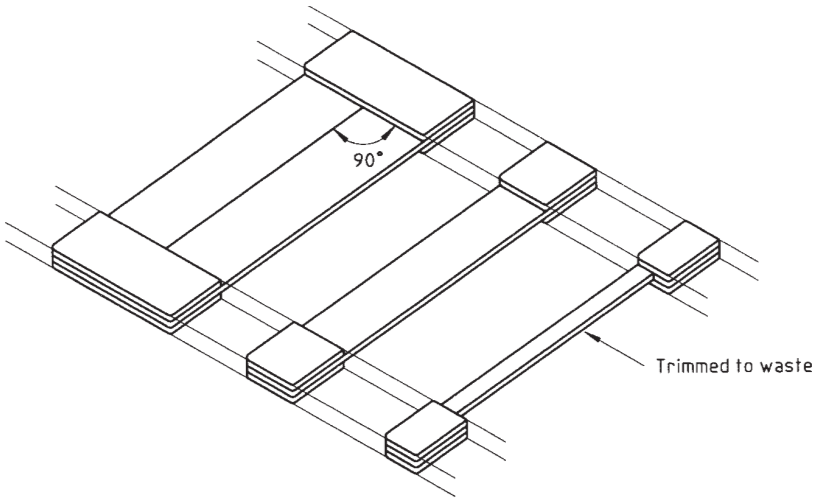


Figure A.1 — Tabbed panel for specimen preparation

Annex B
(informative)

Alignment of specimens

It is recommended that the alignment of the tensile-testing machine and the test specimen be checked at the centre of the gauge length using a strain-gauged coupon of the same material as is to be tested. Use a device or procedure which ensures that specimens are positioned in the grips in a repeatable manner. Strain-gauge the coupon as shown in figure B.1, attaching two gauges (SG1, SG2) to one face of the coupon approximately, one-eighth of the specimen width from the edge and midway between the tabs and attaching a third gauge (SG3) on the centreline of the opposite face also midway between the tabs.

Compare the output of the gauges at the mid-point of the strain range used to measure Young's modulus, i.e. at 0,001 5 for the strain values given in 4.6. Using equations (B.1) and (B.2), calculate the bending strain, expressed as a percentage, in the width (B_b) and thickness (B_h) directions, respectively.

$$B_b = \frac{4|\varepsilon_2 - \varepsilon_1|}{3\varepsilon_{av}} \times 100 \quad \ldots \text{(B.1)}$$

$$B_h = \frac{|\varepsilon_{av} - \varepsilon_3|}{\varepsilon_{av}} \times 100 \quad \ldots \text{(B.2)}$$

where

ε_1, ε_2 and ε_3 are the strains recorded by strain gauges SG1, SG2 and SG3, respectively;

$$\varepsilon_{av} = \left(\frac{\varepsilon_1}{4} + \frac{\varepsilon_2}{4} + \frac{\varepsilon_3}{2} \right)$$

Finally, ensure that the bending strains satisfy the condition given in inequality (B.3):

$$B_b + B_h \leq 3{,}0\ \% \quad \ldots \text{(B.3)}$$

NOTES

1 The use of further strain gauges next to the grips will be necessary to check fully all possible sources of misalignment.

2 The alignment of individual specimens can be checked in the width direction using a clip-on extensometer with a longitudinal-strain output for each edge of the specimen.

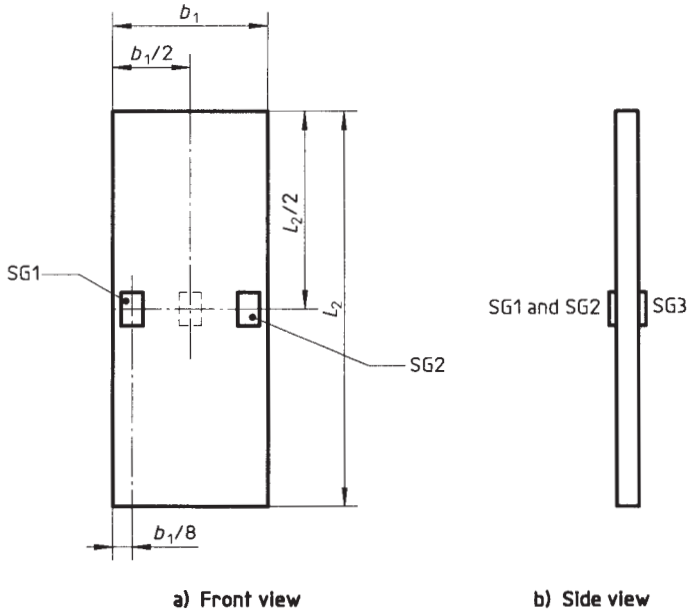


Figure B.1 — Strain-gauge locations (SG1, SG2 and SG3) for system alignment check

Annex ZA (normative)
Normative references to international publications with their relevant European publications

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

Publication	Year	Title	EN	Year
ISO 527-1	1993	Plastics - Determination of tensile properties - Part 1: General principles	EN ISO 527-1	1996
ISO 527-2	1993	Plastics - Determination of tensile - Part 2: Test conditions for moulding and extrusion plastics	EN ISO 527-2	1996
ISO 527-5	1997	Plastics - Determination of tensile - Part 5: Test conditions for unidirectional fibre-reinforced plastic composites	EN ISO 527-5	1997

INTERNATIONAL STANDARD

ISO 14125

First edition
1998-03-01

Fibre-reinforced plastic composites — Determination of flexural properties

Composites plastiques renforcés de fibres — Détermination des propriétés de flexion

Reference number
ISO 14125:1998(E)

Contents

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 14125 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 13, *Composites and reinforcement fibres.*

Annexes A and B form an integral part of this International Standard.

Introduction

This standard is based on ISO 178 but deals with fibre-reinforced plastic composites. As such it retains the test conditions relevant for glass-fibre-reinforced systems. The test conditions are extended from ISO 178 to include both three-point (Method A) and four-point (Method B) loading geometries, and to include conditions for composites based on newer fibres such as carbon and aramid fibres.

Other source documents consulted include ASTM D 790 (four-point loading), prEN 2562 (test conditions), CRAG 200 and JIS K 7074 (use of shims for four-point loading, figure 6). The overall specimen length for four-point loading is the same as for three-point loading.

The scope of ISO 178 will be revised and limited to unreinforced and filled plastics.

EN 63:1977, *Glass-reinforced plastics — Determination of flexural properties — Three-point test*, will be withdrawn.

Fibre-reinforced plastic composites — Determination of flexural properties

1 Scope

1.1 This International Standard specifies a method for determining the flexural properties of fibre-reinforced plastic composites under three-point (Method A) and four-point (Method B) loading. Standard test specimens are defined but parameters included for alternative specimen sizes for use where appropriate. A range of test speeds is included.

1.2 The method is not suitable for the determination of design parameters, but may be used for screening materials, or as a quality-control test.

NOTE – For example, the flexural modulus is only an appropriate value of the tensile Young's modulus of elasticity as the test is not for the additional deflection due to the shear stress which leads to a lower value of the flexural modulus but uses test span/specimen thickness ratios that minimise this effect. Differences between tensile and flexural properties are also caused by the material structure/lay-up.

1.3 The method is suitable for fibre-reinforced thermoplastic and thermosetting plastic composites.

Unreinforced and particle-filled plastics and plastics reinforced with short (i.e. less than 1 mm length) fibres are covered by ISO 178.

1.4 The method is performed using specimens which may be moulded to the chosen dimensions, machined from the central portion of the standard multi-purpose test specimen (see ISO 3167) or machined from semi-finished or finished products such as mouldings or laminates.

1.5 The method specifies preferred dimensions for the specimen. Tests which are carried out on specimens of other dimensions, or on specimens which are prepared under different conditions, may produce results which are not comparable. Other factors, such as the speed of testing and the conditioning of the specimens can influence the results. For materials which are not homogeneous through the section, or above the linear-elastic response region, the result applies only to the thickness and structure tested. Consequently, when comparative data are required, these factors must be carefully controlled and recorded.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possiblitiy of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 178 1993 *Plastics - Determination of flexural properties.*

ISO 291 1997 *Plastics - Standard atmospheres for conditioning and testing.*

ISO 293 1986 *Plastics - Compression moulding test specimens of thermoplastic materials.*

ISO 294-1 1996 *Plastics - Injection moulding of test specimens of thermoplastic materials - Part 1: General principles, and moulding of multipurpose and bar test specimens.*

ISO 295 1991 *Plastics - Compression moulding of test specimens of thermosetting materials.*

ISO 1268 1974 *Plastics - Preparation of glass fibre reinforced, resin bonded, low-pressure laminated plates or panels for test purposes* (under revision).

ISO 2602 1980 *Statistical interpretation of test results - Estimation of the mean - Confidence interval.*

ISO 2818 1994 *Plastics - Preparation of test specimens by machining.*

ISO 3167 1993 *Plastics - Multipurpose test specimens.*

ISO 5893 1993 *Rubber and plastics test equipment - Tensile, flexural and compression types (constant rate of traverse) - Description.*

3 Principle

The test specimen, supported as a beam, is deflected at a constant rate until the specimen fractures or until the deformation reaches some pre-determined value. During this procedure, the force applied to the specimen and the deflection are measured.

The method is used to investigate the flexural behaviour of the test specimens and for determining the flexural strength, flexural modulus and other aspects of the flexural stress/strain relationship under the conditions defined. It applies to a freely supported beam, loaded in three- or four-point flexure. The test geometry is chosen to limit shear deformation and to avoid an interlaminar shear failure.

NOTE – The four-point loading geometry provides a constant bending moment between the central loading members. The compressive contact stresses due to the two central loading members are lower in comparison with the stresses induced under the single loading member of the three-point test. The four-point geometry is chosen so that the centre span equals one-third of the outer span. The distance between the outer support points is the same as in the equivalent three-point loading case, therefore the same specimen can be used.

4 Definitions

For the purpose of this International Standard, the following definitions apply:

4.1 speed of testing, v

The rate of relative movement between the supports and the loading member(s), expressed in millimetres per minute (mm/min).

4.2 flexural stress, σ_f

The nominal stress in the outer surface of the test specimen at mid-span. It is calculated according to the relationship given in clause 10, equation (3) or (8), and is expressed in megapascals (MPa).

4.3 flexural stress at break (rupture), σ_{fB}

The flexural stress at break (or rupture) of the test specimen (see figure 1, curves A and B). It is expressed in megapascals (MPa).

4.4 flexural strength, σ_{fM}

The flexural stress sustained by the test specimen at the maximum load (see figure 1) for acceptable failure modes (see subclause 9.9 and figure 6). It is expressed in megapascals (MPa).

4.5 deflection, s

The distance through which the top or bottom surface of the test specimen at mid-span has deflected during flexure from its original position. It is expressed in millimetres (mm).

4.6 deflection at break, s_B

The deflection at break of the test specimen (see figure 1, curves A and B). It is expressed in millimetres (mm).

4.7 deflection at flexural strength, s_M

The deflection at the load equal to the flexural strength (4.4) (see figure 1, curves A and B). It is expressed in millimetres (mm).

4.8 flexural strain, ε_f

The nominal fractional change in length of an element in the outer surface of the test specimen at mid-span. It is used for calculating the flexural modulus (4.9) and is expressed as a dimensionless ratio.

4.9 modulus of elasticity in flexure; flexural modulus; chord modulus, E_f

The ratio of the stress difference ($\sigma_f'' - \sigma_f'$) divided by the corresponding strain difference ($\varepsilon_f'' = 0{,}0025 - \varepsilon_f' = 0{,}0005$) (see 10.1.2 and 10.2.2). It is expressed in megaspascals (MPa).

NOTE – With computer-assisted equipment, the determination of the modulus using two distinct stress/strain points can be replaced by a linear regression procedure applied to the part of the curve between the two points.

4.10 interlaminar shear modulus, G_{13}

The shear modulus in the through-thickness direction for laminated materials. It is expressed in megapascals (MPa).

NOTE – For materials with mainly in-plane reinforcement, the shear modulus G_{13} is of the order of 3 000 MPa to 6 000 MPa.

4.11 specimen coordinate axes (aligned materials)

The coordinate axes for an aligned material are defined in figure 2. The direction parallel to the fibre axes is defined as the "1" direction and the direction perpendicular to it the "2" direction.

For other materials, the 1, 2 and 3 directions are generally described by the *x*, *y*, *z* system of coordinates.

NOTES

1 The "1" direction is also referred to as the 0 degree (0°) or longitudinal direction, and the "2" direction as the 90 degree (90°) or transverse direction.

2 A similar definition can be used for material with a preferred fibre lay-up or in cases where a direction (e.g. the lengthwise direction) can be related to the production process.

For materials with anisotropy as defined above, the designations include an additional subscript "1" or "2" to indicate the direction tested.

5 Apparatus

5.1 Test machine

5.1.1 General

The test machine shall comply with ISO 5893 as appropriate to the requirements given in 5.1.2 to 5.1.4, as follows:

5.1.2 Speed of testing

The test machine shall be capable of maintaining the speed of testing (4.1), as specified in table 1.

Table 1 – Recommended values for the speed of testing

Speed (mm/min)	Tolerance (%)
0,5	± 20
1	± 20
2	± 20
5	± 20
10	± 20
20	± 10
50	± 10
100	± 10
200	± 10
500	± 10

The speed 0,5 mm/min is not indicated in ISO 5893. The tolerances on the speeds 1 mm/min and 2 mm/min are lower than those indicated in ISO 5893.

5.1.3 Loading member(s) and supports

Supports and central loading member(s) are arranged according to figure 3 (3-point) or figure 4 (4-point). The radius R_1 and the radius R_2 shall be as given in table 2. The axes of the supports and the loading member(s) shall be parallel.

The span L (distance between the supports) shall be adjustable.

Table 2 – Loading and support member dimensions

Dimension	Value (mm)
R_1	5 ± 0,2
R_2 for $h \leq 3$ mm	2 ± 0,2
R_2 for $h > 3$ mm	5 ± 0,2

5.1.4 Load and deflection indicators

The error in the indicated force shall not exceed ± 1 % and that in the indicated deflection shall not exceed ± 1 % of full scale (see ISO 5893).

Deflection obtained from movement of the test machine crosshead shall be corrected for loading train deflection and indentation at the loading points.

5.2 Micrometers and gauges

5.2.1 Micrometer, or equivalent, capable of reading to 0,01 mm or less, and suitable for measuring the width b and thickness h of the test specimen.

The micrometer shall have contact faces appropriate to the surface being measured (i.e. flat faces for flat, polished surfaces and hemispherical faces for irregular surfaces).

5.2.2 Vernier callipers, or equivalent, accurate to within 0,1 % of the span L, for determining the span (see 9.2).

6 Test specimens

6.1 Shape and dimensions

6.1.1 General

Unless otherwise agreed, the dimensions of the specimen shall comply with those given in the standard for the material under test or those given in 6.1.3.

6.1.2 Test direction

The test specimen axis shall be in one of the principal directions (see 4.11 and figure 5).

NOTE – When the material under test shows a significant difference in properties between the two principal directions (i.e. "1" and "2"), it is recommended that testing be carried out in both directions.

If, because of the application, the material is subjected to stress at some specific orientation to the principal directions, the material shall be tested in that orientation. The orientation of the test specimens relative to the principal directions shall be recorded.

6.1.3 Preferred specimen type

Table 3 – Preferred test specimens for method A (three-point flexure)

Dimensions in millimetres

Material	Specimen length (l)	Outer span (L)	Width (b)	Thickness (h)
Class I Discontinuous-fibre-reinforced thermoplastics	80	64	10	4
Class II Plastics reinforced with mats, continuous matting and fabrics, as well as mixed formats (e.g. DMC, BMC, SMC and GMT)	80	64	15	4
Class III Transverse (90°) unidirectional composites; undirectional (0°) and multidirectional composites with $5 < E_{f1}/G_{13} \leq 15$ (e.g. glass-fibre systems)	60	40	15	2
Class IV Unidirectional (0°) and multidirectional composites with $15 < E_{f1}/G_{13} \leq 50$ (e.g. carbon-fibre systems)	100	80	15	2
Tolerances	− 0 + 10	± 1	± 0,5	± 0,2
NOTE – To reduce variability in data for specimens using coarse reinforcements, a specimen width of 25 mm may be used.				

In any one test, the specimen thickness within the central one-third of the length shall nowhere deviate by more than 2 % from the mean value in the central region. The corresponding maximum deviation for width is 3 %. The cross-section shall be rectangular and without rounded edges.

NOTE – The preferred Class I specimen may be machined from the central part of the multipurpose test specimens given in ISO 3167.

Table 4 – Preferred test specimens for method B (four-point flexure)

Dimensions in millimetres

Material	Specimen length (l)	Outer span (L)	Inner span (L')	Width (b)	Thickness (h)
Class I Discontinuous-fibre-reinforced thermoplastics	80	66	22	10	4
Class II Plastics reinforced with mats, continuous matting and fabrics, as well as mixed formats (e.g. DMC, BMC, SMC and GMT)	80	66	22	15	4
Class III Transverse (90°) unidirectional composites; undirectional (0°) and multidirectional composites with $5 < E_{f1}/G_{13} \leq 15$ (e.g. glass-fibre systems)	60	45	15	15	2
Class IV Unidirectional (0°) and multidirectional composites with $15 < E_{f1}/G_{13} \leq 50$ (e.g. carbon-fibre systems)	100	81	27	15	2
Tolerances	$^{+10}_{-0}$	± 1	± 1	± 0,5	± 0,2
NOTE – To reduce variability in the data obtained for specimens using coarse reinforcements, a specimen width of 25 mm may be used.					

In any one test, the specimen thickness over the complete length shall nowhere deviate by more than 2 % from the mean value. The corresponding maximum deviation for width is 3 %. The cross-section shall be rectangular and without rounded edges.

6.1.4 Other test specimens

When it is not possible or desirable to use the preferred test specimen, the dimensions of L, l, h and b in tables A.1 and A.2 in annex A shall apply.

6.2 Specimen preparation

6.2.1 Moulding and extrusion compounds

Specimens shall be prepared in accordance with the relevant material specification. When none exists, or when otherwise specified, specimens shall be either directly compression moulded or directly injection moulded from the material in accordance with ISO 293, ISO 294-1 or ISO 295, as appropriate.

6.2.2 Plates

Specimens shall be machined from plates in accordance with ISO 2818.

6.2.3 Long-fibre-reinforced plastic materials

Specimens shall be machined from a panel prepared in accordance with ISO 1268 or another specified or agreed-upon procedure. Guidance on machining of plastics is given in ISO 2818.

6.3 Checking the test specimens

The specimens shall be free of twist and shall have mutually perpendicular pairs of parallel surfaces. The surfaces and edges shall be free from scratches, pits, sink marks and flashes. The specimens shall be checked for conformity with these requirements by visual observation against straight-edges, squares and flat plates, and by measuring with micrometer callipers. Specimens showing measurable or observable departure from one or more of these requirements shall be rejected or machined to the required size and shape before testing.

7 Number of test specimens

7.1 At least five test specimens giving valid failures shall be tested. The number of measurements may be more than five if greater precision of the mean value is required.

It is possible to evaluate this by means of the confidence interval (95 % probability, see ISO 2602).

7.2 The results from test specimens that rupture outside the central one-third in three-point tests and outside the central portion in four-point tests shall be discarded and new specimens tested in their place.

8 Conditioning

Where applicable, condition the test specimens as specified in the standard for the material under test. In the absence of this information, select the most appropriate conditions from ISO 291, unless agreed otherwise by the interested parties (e.g. for testing at elevated or low temperatures).

9 Procedure

9.1 Where applicable conduct the test in the atmosphere specified in the standard for the material under test. In the absence of this information, select the most appropriate conditions from ISO 291, unless agreed otherwise by the interested parties (e.g. for testing at elevated or low temperatures).

9.2 Measure the width b and the thickness h to the nearest 1 % in the centre of each test specimen. Discard any specimen with a thickness exceeding the tolerance of ± 2 % of the mean value and replace it by another one, selected at random. Calculate the mean thickness h of the set of specimens.

Report if specimens are used that do not meet this thickness tolerance requirement.

Adjust the span L to within 1 % of the calculated value, to comply with the test span/mean specimen thickness ratio L/h given in tables 3 and 4 for preferred specimen sizes, and measure the resulting span to better than 0,2 % of the calculated value.

Tables 3 and 4 shall be used unless unacceptable failures modes (e.g interlaminar shear) are obtained (see figure 6). In this case, a higher value of L/h shall be used. Acceptable ratios are, in order, 16/1, 20/1, 40/1 and 60/1.

9.3 Where applicable, set the speed of testing as given in the standard for the material being tested. In the absence of this information, select the value in table 1 that gives a strain rate as near as possible to 0,01. The speed can be calculated from the following equations:

$$v = \frac{\varepsilon' L^2}{6h} \quad \text{(3-point)} \qquad (1)$$

$$v = \frac{\varepsilon' L^2}{4{,}7h} \quad \text{(4-point)} \qquad (2)$$

where

ε' is a strain rate of 0,01 (i.e. 1 % per minute).

This results in the test speed that produces a deflection closest to 0,4 times the specimen thickness in 1 min, e.g. 2 mm/min for the preferred Class I materials given in 6.1.3.

9.4 Place the test specimen symmetrically on the two supports and identify the tensile face (i.e. the lower face in figures 3 and 4).

9.5 (Optional.) A thin shim or cushion may be placed between the loading member and the specimen to discourage failure of the compressive face of the specimen, in particular for Class III and IV materials.

NOTE – A 0,2 mm thick shim of polypropylene has been found to be successful in reducing failures of the compressive face associated with the loading member.

9.6 Apply the force at mid-span for three-point and equally on both loading members for four-point (see figures 3 and 4).

9.7 Record the force and the corresponding deflection of the specimen during the test, using, if practicable, an automatic recording system that yields a complete flexural load/displacement or flexural stress/flexural strain curve for this operation (see figure 1).

9.8 Determine all relevant stresses, deflections and strains compiled in clause 4 (definitions) from a force/deflection or stress/strain curve or equivalent data.

9.9 Record the type of failure on the basis of figure 6 (indicating tensile or compressive face).

10 Calculation and expression of results

NOTE – Alternative equations are given in annex B to correct for large-deflection effects (i.e. at deflections greater than 0,1 x L mm).

10.1 Method A (three-point flexure)

10.1.1 The flexural stress σ_f is given by the following equation:

$$\sigma_f = \frac{3FL}{2bh^2} \quad (3)$$

where

σ_f is the flexural stress, in megapascals (MPa);

F is the load in newtons (N);

L is the span, in millimetres (mm);

h is the thickness of the specimen, in millimetres (mm);

b is the width of the specimen, in millimetres (mm).

10.1.2 For the measurement of the flexural modulus, calculate the deflections s' and s'', which correspond to the given values of flexural strain $\varepsilon_f' = 0{,}0005$ and $\varepsilon_f'' = 0{,}0025$, by the following equation:

$$s' = \frac{\varepsilon_f' L^2}{6h} \text{ and } s'' = \frac{\varepsilon_f'' L^2}{6h} \quad (4)$$

where

s' and s'' are the beam mid-point deflections, in millimetres (mm);

ε_f' and ε_f'' are the flexural strains, whose values are given above.

The flexural modulus is calculated from equation 5 or 6:

(i) Using equation 5

$$E_f = \frac{L^3}{4bh^3}\left(\frac{\Delta F}{\Delta s}\right) \quad (5)$$

where

E_f is the flexural modulus of elasticity, expressed in megapascals (MPa);

Δs is the difference in deflection between s'' and s';

ΔF is the difference in load F'' and load F' at s'' and s' respectively.

(ii) Using equation 6

$$E_f = 500\left(\sigma_f'' - \sigma_f'\right) \quad (6)$$

where

σ_f' is the stress measured at the deflection s', expressed in megapascals (MPa);

σ_f'' is the stress measured at the deflection s'', expressed in megapascals (MPa).

For computer-assisted equipment, see the note to 4.9.

10.1.3 Calculate the strain in the outer surface of the specimen as follows:

$$\varepsilon = \frac{6sh}{L^2} \tag{7}$$

10.2 Method B - Four point flexure

10.2.1 The flexural stress σ_f is given by the following equation:

$$\sigma_f = \frac{FL}{bh^2} \tag{8}$$

where

σ_f is the flexural stress, in megapascals (MPa);

F is the load, in newtons (N);

L is the span, in millimetres (mm);

h is the thickness of the specimen, in millimetres (mm);

b is the width of the specimen, in millimetres (mm).

10.2.2 For the measurement of the flexural modulus, calculate the deflections s' and s", which correspond to the given values of flexural strain $\varepsilon_f' = 0{,}0005$ and $\varepsilon_f'' = 0{,}0025$, by the following equation:

$$s' = \frac{\varepsilon_f' L^2}{4{,}7\, h} \text{ and } s'' = \frac{\varepsilon_f'' L^2}{4{,}7\, h} \tag{9}$$

where

s' and s'' are the beam mid-point deflections, in millimetres (mm);

ε_f' and ε_f'' are the flexural strains, whose values are given above.

The flexural modulus is calculated from equation 10 or 11:

(i) Using equation 10

$$E_f = \frac{0{,}21L^3}{bh^3}\left(\frac{\Delta F}{\Delta s}\right) \quad (10)$$

where

E_f is the flexural modulus of elasticity, expressed in megapascals (MPa);

Δs is the difference in deflection between s'' and s';

ΔF is the difference in load F'' and load F' at s'' and s' respectively.

(ii) Using equation 11

$$E_f = 500\ (\sigma_f'' - \sigma_f') \quad (11)$$

where

E_f is the flexural modulus of elasticity, expressed in megapascals (MPa);

σ_f' is the stress measured at the deflection s', expressed in megapascals (MPa);

σ_f'' is the stress measured at the deflection s'', expressed in megapascals (MPa).

10.2.3 Calculate the strain in the outer surface of the specimen as follows:

$$\varepsilon = \frac{4{,}7sh}{L^2} \quad (12)$$

For computer-assisted equipment, see the note to 4.9.

10.3 Calculate the arithmetic mean of the individual measurements and, if required, the standard deviation and the 95 % confidence interval of the mean value using the procedure given in ISO 2602.

10.4 Calculate the stresses and the modulus to three significant figures. Calculate the deflections to two significant figures.

11 Precision

The precision of this test method is not known. When inter-laboratory data are obtained, a precision statement will be added at the following revision.

12 Test report

The test report shall include the following information:

a) a reference to this International Standard, indicating the test method, material class and test speed;

b) complete identification of the material tested, including type, source, manufacturer's code number, form and previous history, where these are known;

c) for sheets, the thickness of the sheet and, if applicable, the direction of the major axes of the specimens in relation to some feature on the sheet;

d) the date of measurement;

e) the shape and dimensions of the test specimens (note if the specimens do not meet the thickness tolerance in 9.2);

f) the method of preparing the specimens;

g) the test conditions and conditioning procedures, if applicable;

h) the number of specimens tested;

i) the nominal length of the span used;

j) the speed of testing;

k) the accuracy grading of the test machine (see ISO 5893);

l) the face of the specimen in contact with the loading member(s);

m) the type, material and thickness of the cushion material, if used;

n) the equation used;

o) the test results;

p) the individual measurements, including stress (force) - strain (displacement) diagrams, if required;

q) the type of failure obtained;

r) the standard deviation and the 95 % confidence intervals of the mean values, if required.

Annex A
(normative)

Other test specimens

A.1 The length and thickness of the test specimen shall be in the same ratio as in the preferred test specimen, i.e. as given in table A.1:

Table A.1 – Values for test span *L* and specimen length *l* as a function of thickness *h*

Material class	Three-point		Four-point	
	L/*h*	*l*/*h*	*L*/*h*	*l*/*h*
I	16	20	16,5	20
II	16	20	16,5	20
III	20	30	22,5	30
IV	40	50	40,5	50

unless affected by the provisions of 9.2 (last paragraph).

NOTE – A number of specifications require that test specimens from sheets of thickness greater than a specified upper limit shall be reduced to a standard thickness by machining one face only. In such cases, it is conventional practice to place the test specimen in such a way that the original surface of the specimen is in contact with the two supports and the force is applied by the central loading member(s) to the machined surface of the specimen.

A.2 The applicable value of the width given in table A.2 shall be used.

Table A.2 – Values for width *b* as a function of thickness *h*

Dimensions in millimetres

Nominal thickness *h*	Width (*b*) Class I	Width (*b*) Classes II to IV
$1 < h \leq 3$	25	15
$3 < h \leq 5$	10	15
$5 < h \leq 10$	15	15
$10 < h \leq 20$	20	30
$20 < h \leq 35$	35	50
$35 < h \leq 50$	50	80

For materials with coarse reinforcements, the specimen width shall enable a representative sample to be taken. The tolerances in tables 3 and 4 shall be applied.

Annex B
(normative)

Large-deflection corrections – Calculation and expression of results

B.1 Method A – Three-point flexure

In the case of large deflections, greater than 0,1L, the following equation shall be used for the flexural stress σ_f:

$$\sigma_f = \frac{3FL}{2bh^2}\left\{1+6\left(\frac{s}{L}\right)^2 - 3\left(\frac{sh}{L^2}\right)\right\} \qquad (3a)$$

where

s is the beam mid-point deflection, in millimetres (mm);

σ_f is the flexural stress, in megapascals (MPa);

F is the load, in newtons (N);

L is the span, in millimetres (mm);

h is the thickness of the specimen, in millimetres (mm);

b is the width of the specimen, in millimetres (mm).

And for the strain the following equation shall be used:

$$\varepsilon = \frac{h}{L}\left\{6{,}00\,\frac{s}{L} - 24{,}37\left(\frac{s}{L}\right)^3 + 62{,}17\left(\frac{s}{L}\right)^5\right\} \qquad (7a)$$

The stress is also significantly affected by friction at the loading and support members. This can be solved by placing the members on bearings, by restricting the test method to small deflections (not preferred), or by adding correction terms to equation 3a:

$$\sigma_f = \frac{3FL}{2bh^2}\left\{1+6\left(\frac{s}{L}\right)^2 - 3\left(\frac{sh}{L^2}\right) - \mu\left(2\,\frac{s}{L} - \frac{h}{L}\right)\right\} \qquad (3b)$$

where μ is an effective coefficient of friction that is relatively easy to determine.

B.2 Method B – Four-point flexure

In the case of large deflections, greater than 0,1L, the following equation shall be used for the flexural stress σ_f:

$$\sigma_f = \frac{FL}{bh^2}\left\{1+8{,}78\left(\frac{s}{L}\right)^2 - 7{,}04\left(\frac{sh}{L^2}\right)\right\} \quad (8a)$$

where

σ_f is the flexural stress, in megaspascals (MPa);

F is the load, in newtons (N);

L is the span, in millimetres (mm);

h is the thickness of the specimen, in millimetres (mm);

b is the width of the specimen, in millimetres (mm);

And for the strain the following equation shall be used:

$$\varepsilon = \frac{h}{L}\left\{4{,}70\,\frac{s}{L} - 14{,}39\left(\frac{s}{L}\right)^3 + 27{,}70\left(\frac{s}{L}\right)^5\right\} \quad (11a)$$

Correcting for friction effects as above gives:

$$\sigma_f = \frac{FL}{bh^2}\left\{1+8{,}78\left(\frac{s}{L}\right)^2 - 7{,}04\left(\frac{sh}{L^2}\right) - 3{,}39\,\mu\left(\frac{s}{L}\right)\right\} \quad (11b)$$

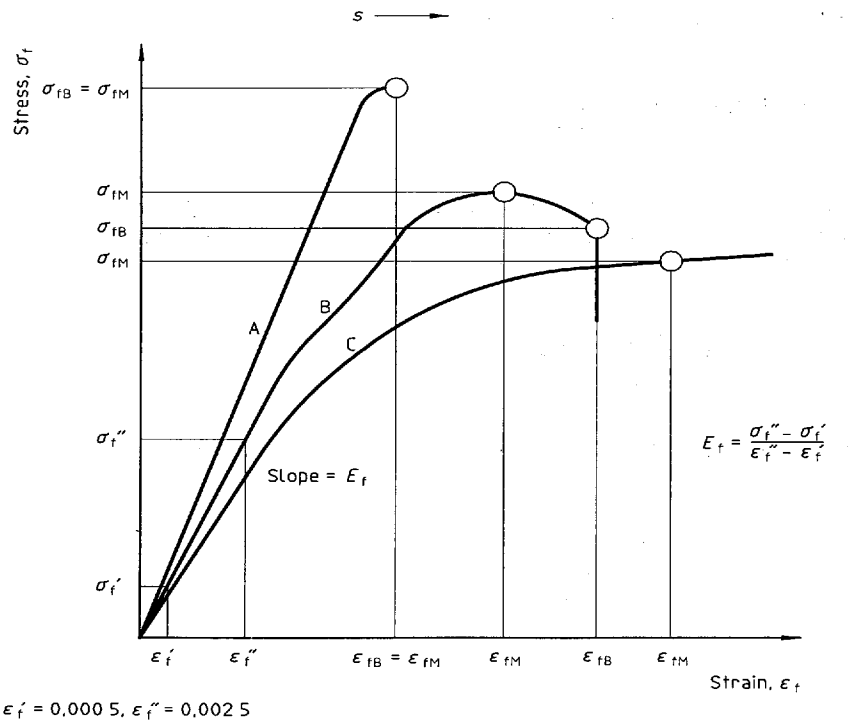

Figure 1 – Typical stress-strain curve
(N.B. Strains ε' and ε'' are equivalent to displacements s' and s'', respectively)

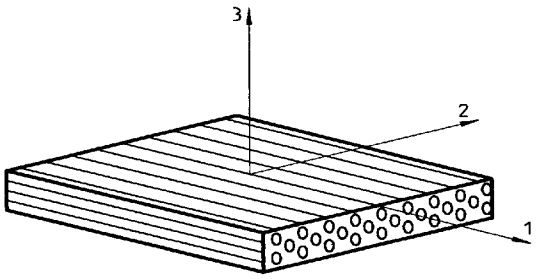

Figure 2 – Unidirectional reinforced composite plate element showing symmetry axes

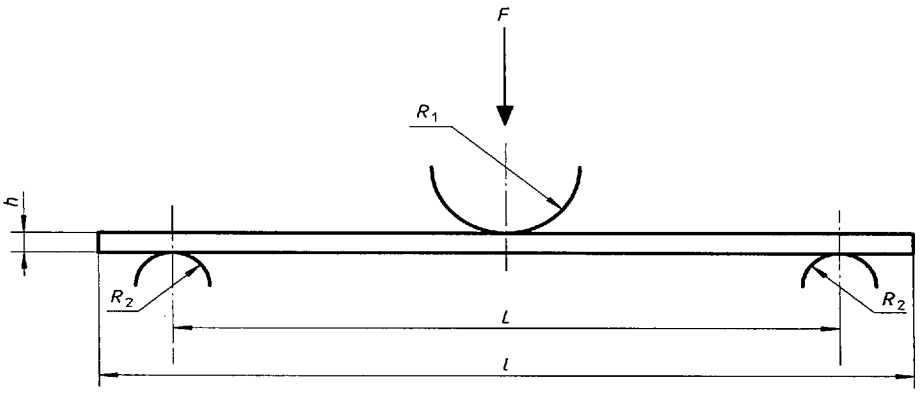

Figure 3 – Three-point loading arrangement

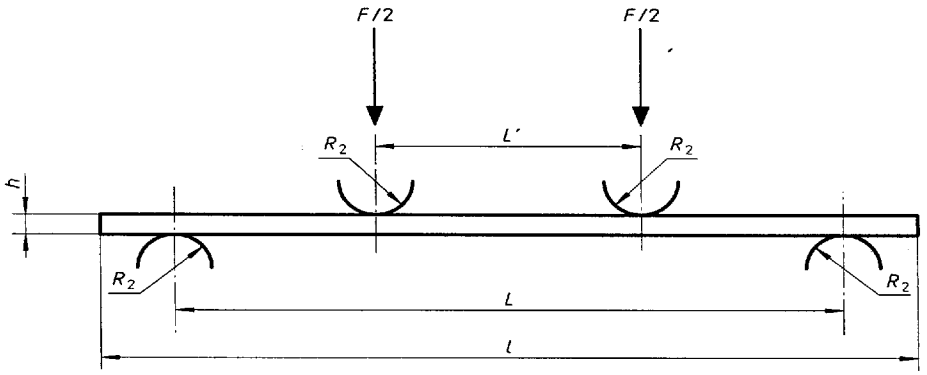

Figure 4 – Four-point loading arrangement
(N.B. $L = 3L'$)

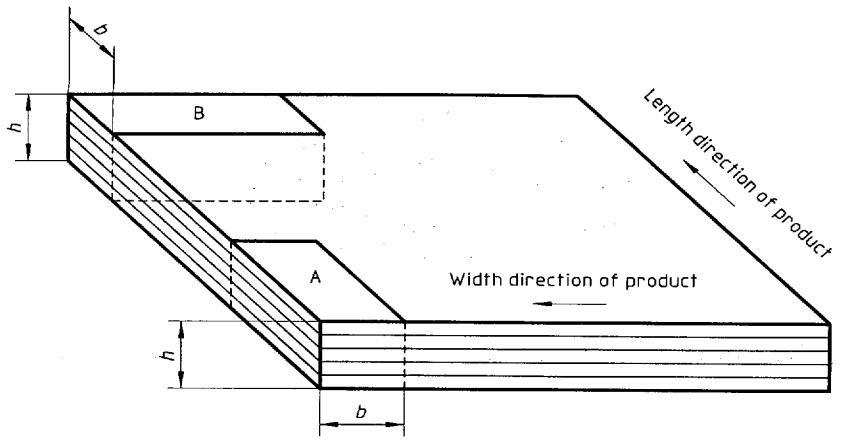

Figure 5 – Location of specimens

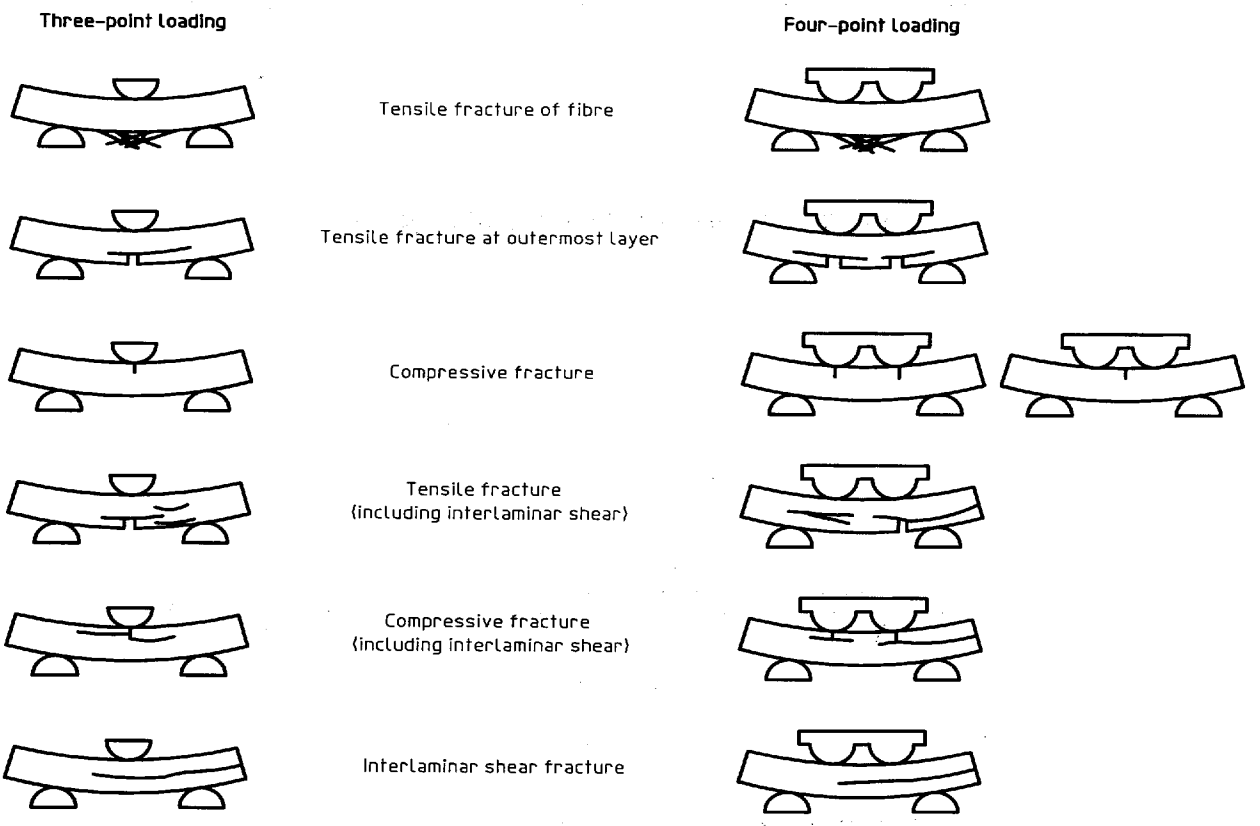

Figure 6 – Examples of possible failure modes
(Tensile-initiated and compression-initiated, remote from the loading points, are acceptable failure modes. Failures initiated by interlaminar shear are not acceptable.)

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